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QUALIFICATION OF ANALYTICAL REFERENCE ENERGETIC MATERIALS

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Secondary explosives	picric acid	Infrared spectroscopy
2,4,6-TNT	High performance liquid chromatography	
RDX	Gas chromatography	
2,4- and 2,6-DNT	Differential scanning calorimetry	
tetryl	Nuclear magnetic resonance	

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Six secondary explosives (2,4,6-TNT, RDX, 2,4- and 2,6-DNT, tetryl, and picric acid) have been qualified as standard analytical references. The instrumental techniques used in this task included gas and high performance liquid chromatography, differential scanning calorimetry, nuclear magnetic resonance, and infrared spectroscopy. Approximately 500-g quantities of each of the standard analytical reference materials are being kept under surveillance at 0°C and are available for distribution upon request.

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FOREWORD

The requirement to determine levels of chemical contamination on, or migrating from, installations is of concern to the Department of the Army, Project Manager for Chemical Demilitarization and Installation Restoration. Therefore, a Quality Control (QC) program has been established at Chemical Systems Laboratory, Aberdeen, Maryland. To implement the QC program, standard explosives are being provided, prepared, and reposited under continuous surveillance by the Large Caliber Weapon Systems Laboratory, Dover, New Jersey. Also, a Standard Analytical Reference Material (SARM) work plan has been formulated involving, initially, six materials. This work plan is funded through the Product Assurance Directorate of ARRADCOM.

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INTRODUCTION

The requirement to determine levels of chemical contamination on, or migrating from, installations necessitates chemical analyses which unequivocally establish these levels. Since the results of these analyses will provide the basis for making critical decisions, it is imperative that the analytical data be valid. Therefore, a Quality Control (QC) program has been established to insure the scientific reliability and compatibility of laboratory data generated in support of the Army Installation Restoration Program. For the implementation of the QC program, standard explosives are being provided, prepared, and reposited under continuous surveillance.

A Standard Analytical Reference Material (SARM) work plan has been formulated involving, initially, TNT, RDX, 2,4-DNT, 2,6-DNT, picric acid, and tetryl. With time, the number of standard explosives will be increased, and involvement will be on a continuing basis. This program entails the procurement and purification of the standards to 98% or better. The purity of the SARM's, as determined by the most modern instrumental methods of analysis directly after purification and after aggravated storage, will be checked periodically while the SARM's are in storage at 0°C.

EXPERIMENTAL

Purification of SARM's

TNT: Procedure as described in reference 1.

RDX: Procedure as given in reference 2.

2,4-DNT: As received, K&K Laboratories, Plainview, New York.

2,6-DNT: As received, K&K Laboratories, Plainview, New York.

Picric acid: Procedure as described in reference 3.

Tetryl: Procedure as given in reference 4.

Record of Identity

Infrared (IR), Perkin-Elmer model 621; procedure as given in reference 8.

Nuclear Magnetic Resonance (NMR), Varian model T60; procedure as described in reference 7.

Aggravated Storage Test

Approximately 2 g of each SARM contained in screw-top glass bottles were stored for a period of 2 weeks in a constant temperature chamber set at 70°C ± 1°C. The accelerated thermal effect on the stability of the SARM's was determined by Differential Scanning Calorimetry (DSC), and checked by High Performance Liquid Chromatography (HPLC), or Gas Chromatography (GC), before and after storage at 70°C.

Purity Determination

Equipment: Perkin-Elmer model IB DSC. Special conditions: sample size, between 1 and 2 mg; heating rate, 1.25°C/minute; range, 1; chart speed, 160 mm/minute.

Procedure as described in reference 5.

The SARM's of acceptable purity were stored at 0°C ± 1°C.

Elemental Analysis

Samples of SARM's were submitted to Schwarzkopf Microanalytical Laboratory, Woodside, New York, for C, H, and N determinations before storage at 70°C. All determinations were run in duplicate.

Fingerprints

Gas Chromatography: Hewlett Packard model 7626A; procedure as described in reference 6.

High Performance Liquid Chromatography: Perkin-Elmer Series 3; procedures as stipulated in respective figures.

RESULTS AND DISCUSSION

RECORD OF IDENTITY OF SARM'S

The infrared spectra of Standard Analytical Reference (SAR) 2,4,6-TNT, RDX, 2,4-DNT, 2,6-DNT, picric acid (PA), and tetryl shown in figures 1 through 6, respectively, were compared to those reported by Pristera, et al., (ref 9) and found to be identical in every specific absorbance frequency.

As an independent and complimentary method of identification, NMR spectra of the above SARM's were also generated and compared to those published by Hogan and Richter (ref 7). The NMR spectrum (fig. 7) and its integral (fig. 8) obtained for SAR-TNT dissolved in acetone confirms its identity. The line positions for the three methyl and two ring protons agree within experimental error with the published spectrum (fig. 9). As expected, the ratio of the respective peak areas obtained from the integral of the spectrum, within experimental error, is 3:2.

The NMR spectra obtained for SAR-RDX (fig. 10) and SAR 2,4-DNT (fig. 11) confirm the identities of these materials. The line positions seen in these spectra agree with those of the published spectra. The integral (fig. 12) of the 2,4-DNT spectrum shows the expected peak area ratios (1:1:1:3) for the three ring protons and the methyl protons.

The only published NMR spectrum of 2,6-DNT in reference 7 is for a sample dissolved in thionyl chloride ($SOCl_2$), a solvent which is currently unavailable. However, the line pattern seen in the published spectrum is repeated in the spectra obtained with d-acetone (fig. 13) and d-DMSO (fig. 14) although the ring proton signals are shifted downfield somewhat. The line position data are summarized in table 1. Individual line assignments for the ring protons have not been made. However, the integral of the d-acetone spectrum (fig. 15) shows that the downfield ring proton pattern represents the same number of protons as the three methyl proton signal at approximately 150 Hz. The data above lend credence to establishing the identity of the sample as 2,6-DNT.

Identification of SAR-PA by NMR is less straightforward. In d-acetone solution the position of the signal attributable to the labile phenolic protons (fig. 16) has been found to vary with each type of solvent (ref 7). In d-DMSO solution (fig. 17) the phenolic proton apparently reacts with water normally present in the solvent, causing the water line at 200 Hz to disappear and changing the position, shape, and amplitude of its own line. The integral of the d-acetone spectrum (fig. 18) shows the expected ratio of approximately 2:1 for the ring and phenolic proton signals, respectively. The line position data are summarized in table 2 (ref 10). This supportive evidence corroborates the sample as being PA.

The NMR spectrum (fig. 19) and integral (fig. 20) obtained for SAR-tetryl dissolved in d-acetone confirm its identity. The line positions for the three methyl and two ring protons agree within experimental error with those published in reference 7. As expected, the ratio of the respective peak areas, within experimental error, is 3:2.

For the record, under the category of identification and in support of the more absolute methods cited above, elemental analyses of the SARM's were conducted prior to storage at 70°C and the results obtained are listed in table 3. The values are shown to be within one percent of those calculated.

Purity of SARM's

The detailed data on the purity of the SARM's, prior to aggravated storage, as determined by DSC, are listed in tables 4 through 9, and purity data of the SARM's after aggravated storage are shown in tables 10 through 15. For convenience of comparative evaluation of the stability of the SARM's as a result of aggravated storage at 70°C, the respective pre- and post-storage percent purity values are listed in table 16. All of the SAR samples are shown to be of acceptable purity and thermal stability under the conditions of the ten replicate determinations. The percent changes in purity are within the reported experimental error of ± 0.045 percent (ref 5), except for the SAR's 2,4- and 2,6-DNT. Their respective purities are observed to have increased as a result of storage at 70°C over a 2-week period. Apparently, the trace impurities present initially in the SARM's are volatile and hence are driven off, rendering the host materials purer to the extents shown.

Fingerprinting of SARM's

The fingerprinting of the SARM's has been included in this qualification program as a complementary method to the DSC for purity determination. The fingerprinting by GC for volatile and HPLC for non-volatile SARM's is used to profile impurities before and after aggravated storage. For the surveillance testing every 6 months, the fingerprinting will be used in conjunction with the DSC determinations in order to detect trends in impurity profiles.

The impurity profiles of TNT determined by GC before and after aggravated storage are shown in figures 21 and 22, respectively. The impurities identified are present at trace levels (less than 10 ppm) and, therefore, are expected to have a minor effect on the purity as indicated above by DSC. What is of noteworthy interest is the fact that the impurity peaks numbered 3,4,and 5 changed only slightly in intensity, and that no new impurities were generated after storage at 70°C.

The SAR-RDX, because of its relative nonvolatility, was run on the HPLC before and after 2 weeks storage at 70°C (fig. 23). The only impurity peak observed is due to HMX and its magnitude corresponds to the amount indicated by the DSC percent purity determinations. The aggravated storage test induced no change in the impurity profile.

The GC impurity profiles of SAR-2,4-DNT before and after storage are shown in figures 24 and 25. The unknown impurity designated by 3 is apparently associated in part with purity values obtained by DSC (table 16). The volatile impurity mentioned earlier might be related to the percent change corresponding to 15 ppm. The disappearance of the volatile impurity is not reflected by the chromatograms, probably due to the fact that the retention time of the impurity is identical with that of the solvent acetone. A small unsymmetrical peak at 17.6 minutes is observed in figure 25 which may indicate the formation of an impurity during the storage test.

The GC impurity profile (fig. 26) of SAR-2,6-DNT, before the storage test, depicts a trace component designated 4 at approximately 17 minutes retention time. This impurity does not change in intensity after the storage test (fig. 27). On the other hand, a new impurity peak appears at 18 minutes, apparently resulting from the aggravated storage test. As in the case of the 2,4-DNT, the disappearance of the volatile component in figure 26 may also be attributed to a similar retention time with the solvent peak.

Attempts to chromatograph SAR-PA and tetryl on the GC were unsuccessful. Therefore, both were run on the HPLC as shown in figures 28 and 29. These chromatograms are devoid of any impurities as indicated by HPLC. The aggravated storage test had no effect on the purity profile of either of these SARM's.

CONCLUSIONS

The secondary explosives TNT, RDX, 2,4-DNT, 2,6-DNT, PA, and tetryl selected for qualification as SARM's have met all of the requirements prescribed under the established Quality Control Program. The SARM's are presently in a repository at 0°C ± 1°C in approximately 500-g quantities, to be tested for purity every 6 months.

REFERENCES

1. W.R. Tomlinson, Jr. Revised by O.E. Sheffield, "Properties of Explosives of Military Interest, Rev. 1," Picatinny Arsenal Technical Report 1740, Dover, NJ, April 1958.

2. Military Standard Specification MIL-H-45444B, "HMX".

3. Military Standard Specification JAN-A-187, "Acid, Picric (trinitrophenol)".

4. Military Standard Specification MIL-T-339C "Tetryl (trinitrophenylmethylnitramine)".

5. C. Plato and A.R. Glasgow, Jr., "Differential Scanning Calorimetry as a General Method for Determining the Purity and Heat of Fusion of High-Purity Organic Chemicals. Application to 95 Compounds." Anal Chem Vol 41 1969, p330.

6. C. Ribaudo and J. Haberman, "Origin - Identification of Explosives Via Their Composite Impurity Profiles. 1. The relation of the Origin of Military Grade TNT to its Mono-, Di-, and Tri-nitrotoluene Isomer Impurities." Picatinny Arsenal Technical Report 4768, Dover, NJ, October 1975.

7. V.D. Hogan and T. Richter, "A New Convenient Tool for Identifying Composite Explosives: Proton Magnetic Resonance Fingerprinting," Picatinny Arsenal Technical Report 4790, Dover, NJ, June 1975.

8. F. Pristera and W. Fredericks, "Compilation of Infrared Spectra of Ingredients of Propellants and Explosives," Picatinny Arsenal Technical Memorandum 1887, Dover, NJ, September 1959.

9. F. Pristera, M. Halik, A. Castelli, and W. Fredericks, "Analysis of Explosives Using Infrared Spectroscopy," Anal Chem Vol 32, 1960, pp 495-508.

10. T.A. Richter, M. Warman, and E. Sowinski, "NMR Spectral Atlas of Aromatic Nitro Compounds," Picatinny Arsenal Technical Report 3529, Dover, NJ, November 1967.

Table 1. Shifts in proton line positions of 2,6-DNT as a function of solvent

Assignments	Line position (Hz) SOCl (ref 10)	d-acetone (fig. 13)	d-DMSO (fig. 14)
3 methyl protons	151	154	150
3 ring protons	438-482	460-500	459-501

Table 2. Shifts in phenolic proton line positions of PA as a function of solvent

Assignments	Line position (Hz) SOCl (ref 10)	d-acetone (ref 7)	d-acetone (fig. 17)	d-DMSO (fig. 18)
2 ring protons	548	550	549	521
phenolic proton	713	634	618	601
		533		
		662		

Table 3. Elemental analysis of SARM's

SARM	Carbon Found	Carbon Calculated	Hydrogen Found	Hydrogen Calculated	Nitrogen Found	Nitrogen Calculated
2,4,6-TNT	37.09	37.01	2.19	2.19	18.35	18.50
RDX	16.30	16.22	2.70	2.72	37.55	37.84
PA	31.27	31.45	1.37	1.32	18.40	18.34
2,4-DNT	46.16	46.16	3.20	3.32	15.30	15.38
2,6-DNT	46.08	46.16	3.36	3.32	15.31	15.38
Tetryl	29.21	29.28	1.71	1.76	24.04	24.39

Table 4. Purity of TNT prior to aggravated storage

	T_1 (K)	A_1 (g)	T_2 (K)	A_2 (g)	T_3 (K)	A_3 (g)	A (g)	Mole Purity %	Date tested
A	347.594	0.06865	347.676	0.17424	347.729	0.25528	0.56764	98.84	10/26/78
B	347.590	0.07974	347.626	0.20088	347.723	0.29357	0.59030	99.51	10/26/78
C	347.664	0.05828	347.736	0.17852	347.783	0.27620	0.59804	99.20	10/27/78
D	347.693	0.13195	347.717	0.22210	347.747	0.32972	0.80796	99.72	10/27/78
E	347.684	0.10580	347.720	0.17625	347.762	0.27625	0.56837	99.40	10/27/78
F	347.646	0.06328	347.682	0.11885	347.720	0.19246	0.55620	99.60	10/27/78
G	347.634	0.08396	347.666	0.15380	347.698	0.24819	0.68840	99.83	10/27/78
H	347.666	0.12524	347.691	0.20768	347.720	0.32505	0.73724	99.62	10/27/78
I	347.661	0.09447	347.692	0.17093	347.729	0.27618	0.69246	99.32	10/27/78
J	347.577	0.08080	347.609	0.14758	347.639	0.23988	0.72312	99.85	10/27/78
							AVERAGE	99.44	

Table 5. Purity of RDX prior to aggravated storage

	T_1 (K)	A_1 (g)	T_2 (K)	A_2 (g)	T_3 (K)	A_3 (g)	A (g)	Mole purity %	Date tested
A	478.417	0.13710	478.484	0.28784	478.515	0.40837	0.70140	99.87	10/27/78
B	477.446	0.07280	477.632	0.17175	477.726	0.30561	0.69780	99.65	10/31/78
C	478.356	0.08344	478.443	0.18141	478.516	0.37661	0.76704	99.82	10/27/78
D	477.197	0.11265	477.318	0.21483	477.432	0.37475	0.75252	99.71	11/01/78
E	478.262	0.12994	478.427	0.25155	478.509	0.42086	0.99585	99.91	11/02/78
F	477.918	0.13702	478.087	0.25902	478.173	0.41628	0.84445	99.96	11/03/78
G	477.748	0.11365	477.909	0.22239	477.967	0.40090	0.86460	99.96	11/03/78
H	477.874	0.15318	478.127	0.30686	478.256	0.57795	1.17649	99.90	11/02/78
I	477.628	0.09588	477.726	0.19055	477.773	0.27628	0.54487	99.83	11/01/78
J	478.313	0.14962	478.464	0.29733	478.543	0.49590	1.08309	99.90	11/02/78
							Average	99.84	

Table 6. Purity of 2,4-DNT prior to aggravated storage

	T_1 (K)	A_1 (g)	T_2 (K)	A_2 (g)	T_3 (K)	A_3 (g)	A_T (g)	Mole purity (%)	Date tested
A	332.639	.13242	333.031	.21524	333.326	.33855	.79042	99.47	11/13/78
B	332.311	.11635	332.766	.22329	333.218	.39921	.84386	98.65	11/13/78
C	333.089	.11561	333.378	.19635	333.672	.33530	.86964	99.34	11/13/78
D	333.256	.20854	333.684	.41186	333.816	.52417	1.08669	99.38	11/13/78
E	333.825	.15825	334.433	.31441	334.730	.44268	.90595	98.75	11/29/78
F	333.620	.14895	334.220	.32655	334.517	.48236	1.00923	98.93	11/30/78
G	333.801	.14374	334.261	.25725	334.621	.42884	.88178	99.22	11/30/78
H	333.869	.17735	334.436	.33084	334.710	.46664	1.01650	99.14	11/30/78
I	333.300	.20448	333.878	.32588	334.341	.49870	1.03305	98.84	11/30/78
J	333.837	.19120	334.397	.37308	334.576	.47428	.99538	99.13	11/30/78
							AVERAGE	99.09	

Table 7. Purity of 2,6-DNT prior to aggravated storage

	T_1 (K)	A_1 (g)	T_2 (K)	A_2 (g)	T_3 (K)	A_3 (g)	A_T (g)	Mole purity (%)	Date tested
A	328.309	.11382	328.577	.24181	328.699	.35768	.79773	99.62	11/14/78
B	328.746	.08065	329.347	.23596	329.470	.30418	.61585	99.37	11/15/78
C	328.736	.13781	329.000	.28016	329.120	.40187	.82067	99.56	11/15/78
D	328.773	.12414	329.029	.29648	329.144	.43982	.86837	99.53	11/15/78
E	329.810	.13970	330.087	.26793	330.223	.36689	.87610	99.30	11/15/78
F	328.797	.11367	329.100	.19378	329.388	.31392	.76995	99.01	11/15/78
G	329.309	.13528	329.601	.24521	329.729	.32935	.72672	99.47	11/15/78
H	327.912	.12355	328.352	.24715	328.555	.36561	.78780	99.45	11/15/78
I	327.774	.11514	328.220	.21591	328.513	.33858	.75075	99.18	11/15/78
J	328.436	.11042	329.031	.24850	329.243	.35320	.76055	99.33	11/30/78
							AVERAGE	99.38	

Table 8. Purity of picric acid prior to aggravated storage

	T_1 (K)	A_1 (g)	T_2 (K)	A_2 (g)	T_3 (K)	A_3 (g)	A_T (g)	Mole purity (%)	Date tested
A	391.908	.08431	392.065	.21210	392.114	.28810	.62805	99.81	11/15/78
B	391.752	.08501	391.911	.20308	391.950	.28005	.63520	99.91	11/15/78
C	391.844	.08516	392.024	.19115	392.072	.25391	.57257	99.86	11/15/78
D	392.111	.10790	392.232	.18849	392.338	.32034	.63815	99.76	11/15/78
E	391.836	.09698	391.938	.18827	391.973	.26368	.60732	99.93	11/15/78
F	391.817	.10562	391.916	.19615	391.953	.27287	.66180	99.92	11/15/78
G	391.924	.11935	392.023	.22216	392.070	.30320	.62160	99.79	11/15/78
H	392.000	.11477	392.156	.25002	392.191	.33370	.71276	99.85	11/15/78
I	391.841	.11605	391.993	.27425	392.040	.36958	.75873	99.81	11/15/78
J	391.779	.12295	391.863	.23985	391.906	.33668	.66274	99.80	11/15/78
							AVERAGE	99.84	

Table 9. Purity of tetryl before storage

	T_1 (K)	A_1 (g)	T_2 (K)	A_2 (g)	T_3 (K)	A_3 (g)	A_T (g)	Mole purity (%)	Date tested
A	401.650	.12920	401.710	.20259	401.778	.30635	.82455	99.35	1/18/79
B	401.660	.12736	401.799	.26750	401.853	.38285	.89327	99.83	1/18/79
C	401.611	.10316	401.660	.18368	401.698	.30785	.69555	99.91	1/18/79
D	401.981	.09710	402.115	.22210	402.177	.31881	.86686	99.71	1/18/79
E	401.646	.10075	401.778	.23120	401.837	.33550	.82397	99.75	1/18/79
F	401.845	.13745	401.902	.22449	401.958	.34369	.84407	99.67	1/18/79
G	401.630	.14775	401.775	.27450	401.845	.37006	.80942	99.55	1/18/79
H	401.607	.11772	401.738	.25415	401.800	.36339	.88372	99.71	1/18/79
I	401.337	.13048	401.469	.27685	401.535	.38933	.84251	99.55	1/18/79
J	401.559	.08290	401.672	.21620	401.727	.33785	.81050	99.80	1/18/79
							Average	99.68	

Table 10. Purity of TNT after aggravated storage

	T_1 (K)	A_1 (g)	T_2 (K)	A_2 (g)	T_3 (K)	A_3 (g)	A_T (g)	Mole purity (%)	Date tested
A	349.068	.09355	349.123	.16834	349.183	.27965	.67025	99.57	12/6/78
B	349.350	.14571	349.417	.24020	349.478	.35928	.70555	99.52	12/6/78
C	349.140	.12667	349.194	.21530	349.243	.33907	.68679	99.74	12/6/78
D	349.008	.08301	349.103	.25440	349.161	.39500	.79534	99.27	12/6/78
E	349.012	.10940	349.069	.19525	349.115	.32095	.66220	99.84	12/6/78
F	349.098	.11764	349.146	.21620	349.203	.35370	.72581	99.02	12/6/78
G	349.184	.12486	349.230	.22345	349.286	.36020	.70812	98.73	12/6/78
H	349.166	.10166	349.204	.19780	349.249	.33957	.72070	99.47	12/6/78
I	349.157	.13162	349.203	.23589	349.244	.38905	.79145	99.81	12/6/78
J	349.195	.11105	349.246	.20915	349.284	.35121	.71820	99.87	12/6/78
							AVERAGE	99.48	

Table 11. Purity of RDX after aggravated storage

	T_1 (K)	A_1 (g)	T_2 (K)	A_2 (g)	T_3 (K)	A_3 (g)	A_T (g)	Mole purity (%)	Date tested
A	479.398	.17900	479.638	.33007	479.689	.42265	1.05101	99.92	12/08/78
B	479.885	.23292	479.996	.37443	480.027	.50375	.98252	99.93	12/08/78
C	479.071	.11292	479.182	.22945	479.225	.34495	.72410	99.93	12/11/78
D	478.672	.19728	478.899	.34842	478.951	.44638	1.06690	99.92	12/11/78
E	480.444	.23817	480.604	.32575	480.660	.39137	1.06742	99.92	12/11/78
F	479.047	.14827	479.254	.30428	479.296	.42445	.87605	99.94	12/12/78
G	478.908	.19090	479.075	.28885	479.137	.36533	.94924	99.89	12/11/78
H	478.958	.15691	479.214	.26361	479.344	.41209	.83914	99.86	12/13/78
I	478.845	.16001	479.012	.23748	479.120	.39122	.81777	99.92	12/13/78
J	480.128	.15992	480.284	.25487	480.438	.41103	.82776	99.63	12/11/78
							AVERAGE	99.89	

Table 12. Purity of 2,4-DNT after aggravated storage

	T_1 (K)	A_1 (g)	T_2 (K)	A_2 (g)	T_3 (K)	A_3 (g)	A_T (g)	Mole purity (%)	Date tested
A	335.228	.27725	335.652	.38762	335.922	.51450	1.12823	99.37	12/19/78
B	335.555	.22803	335.923	.34233	336.191	.47435	.97838	99.05	12/19/78
C	336.666	.24416	337.012	.41220	337.169	.56005	1.14333	99.56	12/22/78
D	334.306	.21940	334.858	.40124	335.036	.50650	1.02303	99.21	12/20/78
E	334.473	.26425	334.839	.40147	335.100	.57380	1.21395	99.31	12/20/78
F	334.367	.23515	334.740	.35711	335.004	.51346	1.14040	99.38	12/20/78
G	334.037	.16880	334.603	.31915	334.873	.45069	.97330	99.15	12/20/78
H	334.039	.21775	334.410	.33464	334.680	.47214	1.03442	99.12	12/20/78
I	333.825	.20663	334.192	.33227	334.461	.48055	1.02268	99.08	12/20/78
J	335.053	.19231	335.431	.30001	335.702	.42637	.96865	99.17	12/19/78
							AVERAGE	99.24	

Table 13. Purity of 2,6-DNT after aggravated storage

	T_1 (K)	A_1 (g)	T_2 (K)	A_2 (g)	T_3 (K)	A_3 (g)	A_T (g)	Mole purity (%)	Date tested
A	331.459	.16455	331.628	.25218	331.773	.39570	.88162	99.70	12/22/78
B	330.516	.22843	330.592	.29215	330.655	.38022	.92813	99.84	12/22/78
C	330.437	.16902	330.591	.27606	330.656	.35801	.88925	99.79	12/22/78
D	330.361	.22610	330.519	.33314	330.594	.40805	.90468	99.53	12/22/78
E	330.286	.18701	330.434	.30455	330.505	.39405	.92178	99.66	12/22/78
F	330.222	.27582	330.376	.38968	330.453	.47214	1.09120	99.51	12/22/78
G	329.750	.16682	330.076	.35085	330.165	.43414	.88271	99.33	12/22/78
H	329.402	.13898	329.753	.25861	329.981	.46291	.93325	99.67	12/22/78
I	329.487	.19130	329.655	.27777	329.811	.41330	.95970	99.59	12/22/78
J	329.517	.17005	329.840	.37814	329.917	.46780	.95198	99.49	12/22/78
							AVERAGE	99.61	

Table 14. Purity of picric acid after aggravated storage

	T_1 (K)	A_1 (g)	T_2 (K)	A_2 (g)	T_3 (K)	A_3 (g)	A_T (g)	Mole purity (%)	Date tested
A	392.713	.09490	392.840	.19886	392.890	.30305	.71360	99.92	12/14/78
B	392.819	.10596	392.892	.15757	392.961	.22904	.53678	99.74	12/14/78
C	392.953	.11006	393.020	.15420	393.111	.24268	.59315	99.72	12/14/78
D	392.691	.09100	392.836	.19142	392.890	.28202	.62154	99.90	12/14/78
E	392.836	.12595	392.964	.25270	393.026	.35990	.70645	99.76	12/14/78
F	392.798	.10445	392.941	.20600	393.008	.28915	.65268	99.77	12/14/78
G	393.395	.11498	393.536	.22628	393.605	.31295	.62892	99.71	12/14/78
H	392.840	.11635	393.009	.19650	393.078	.26254	.59368	99.83	12/15/78
I	393.199	.11268	393.341	.19690	393.395	.27831	.63332	99.92	12/15/78
J	393.883	.08543	393.057	.14655	393.209	.25772	.55325	99.76	12/15/78
							AVERAGE	99.80	

Table 15. Purity of tetryl after aggravated storage

	T_1 (K)	A_1 (g)	T_2 (K)	A_2 (g)	T_3 (K)	A_3 (g)	A_T (g)	Mole purity (%)	Date tested
A	402.474	.08845	402.623	.20037	402.688	.28332	.67924	99.67	1/18/79
B	401.943	.08110	402.069	.19960	402.126	.31384	.73455	99.84	1/18/79
C	401.980	.08219	402.128	.19688	402.179	.28885	.62180	99.84	1/18/79
D	402.098	.10510	402.106	.18430	402.152	.29890	.66996	99.86	1/18/79
E	402.011	.09780	402.071	.16935	402.118	.27198	.61122	99.86	1/18/79
F	401.924	.07494	402.032	.21125	402.091	.33122	.75358	99.67	1/18/79
G	401.865	.09648	401.973	.23947	402.091	.35650	.78056	99.15	1/18/79
H	402.234	.09824	402.368	.24142	402.427	.35627	.75025	99.72	1/18/79
I	402.012	.13530	402.064	.23102	402.117	.36218	.80995	99.60	1/18/79
J	402.000	.07428	402.062	.13585	402.112	.23170	.77326	99.91	1/18/79
							Average	99.71	

Table 16. Purity of SARM's before and after aggravated storage at 70°C

SARM	Mole purity (%) Prestorage	Poststorage	Percent storage
2,4,6-TNT	99.44	99.48	+ 0.04
RDX	99.84	99.89	+ 0.05
2,4-DNT	99.09	99.24	+ 0.15
2,6-DNT	99.38	99.61	+ 0.23
PA	99.84	99.80	- 0.04
Tetryl	99.68	99.71	+ 0.03

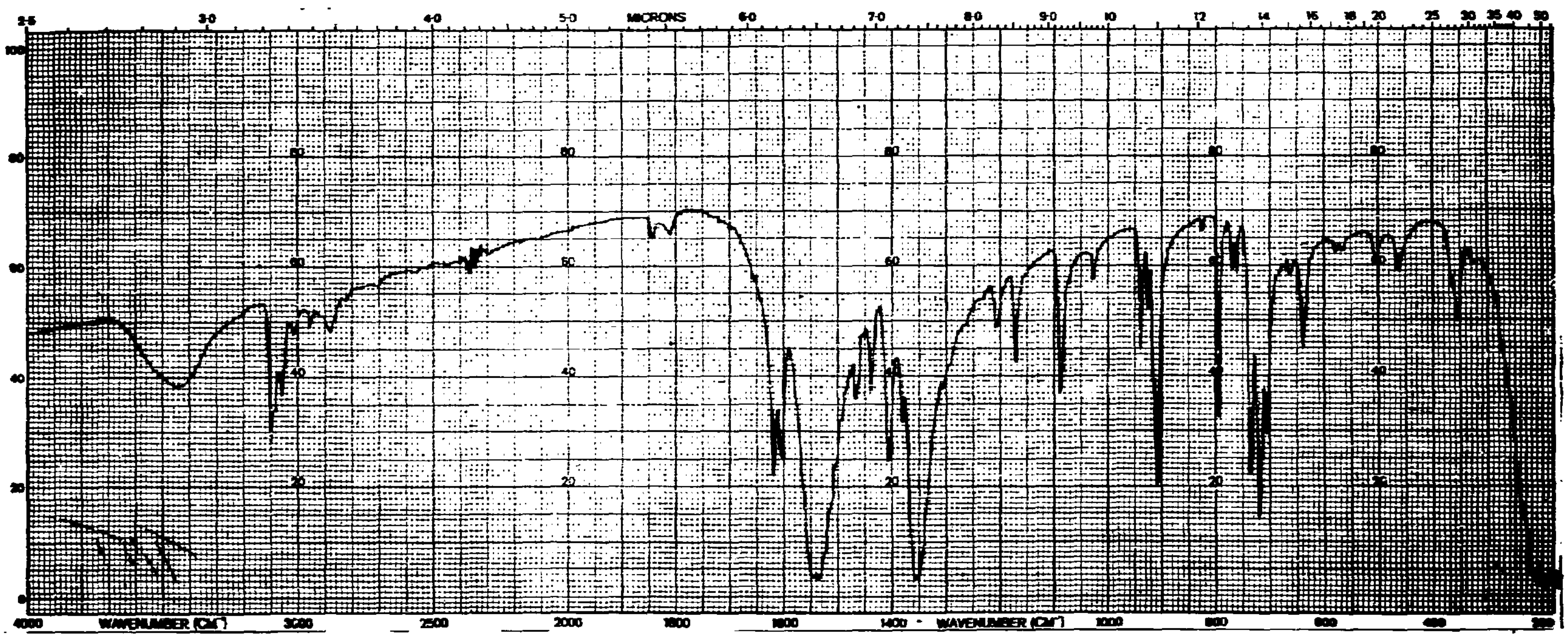


Figure 1. Infrared spectrum of SAR-TNT of 99.44% purity.

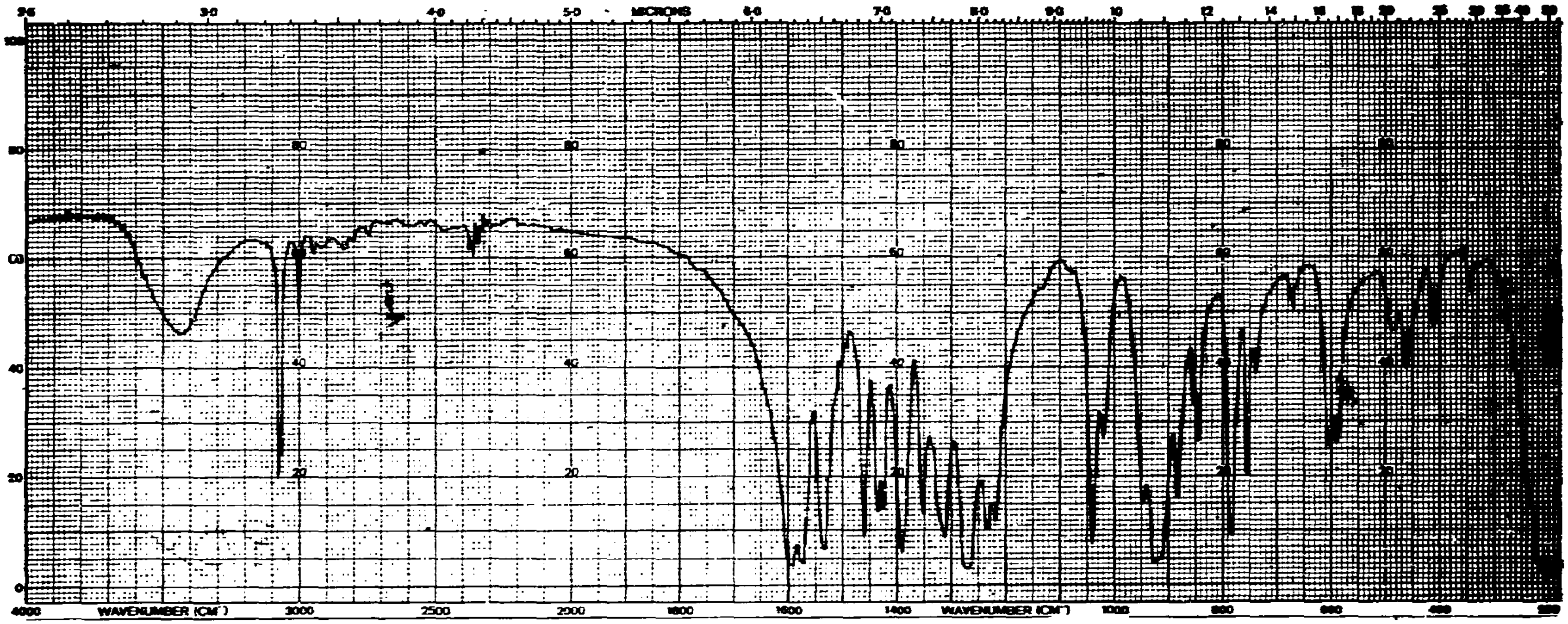


Figure 2. Infrared spectrum of SAR-RDX of 99.84% purity.

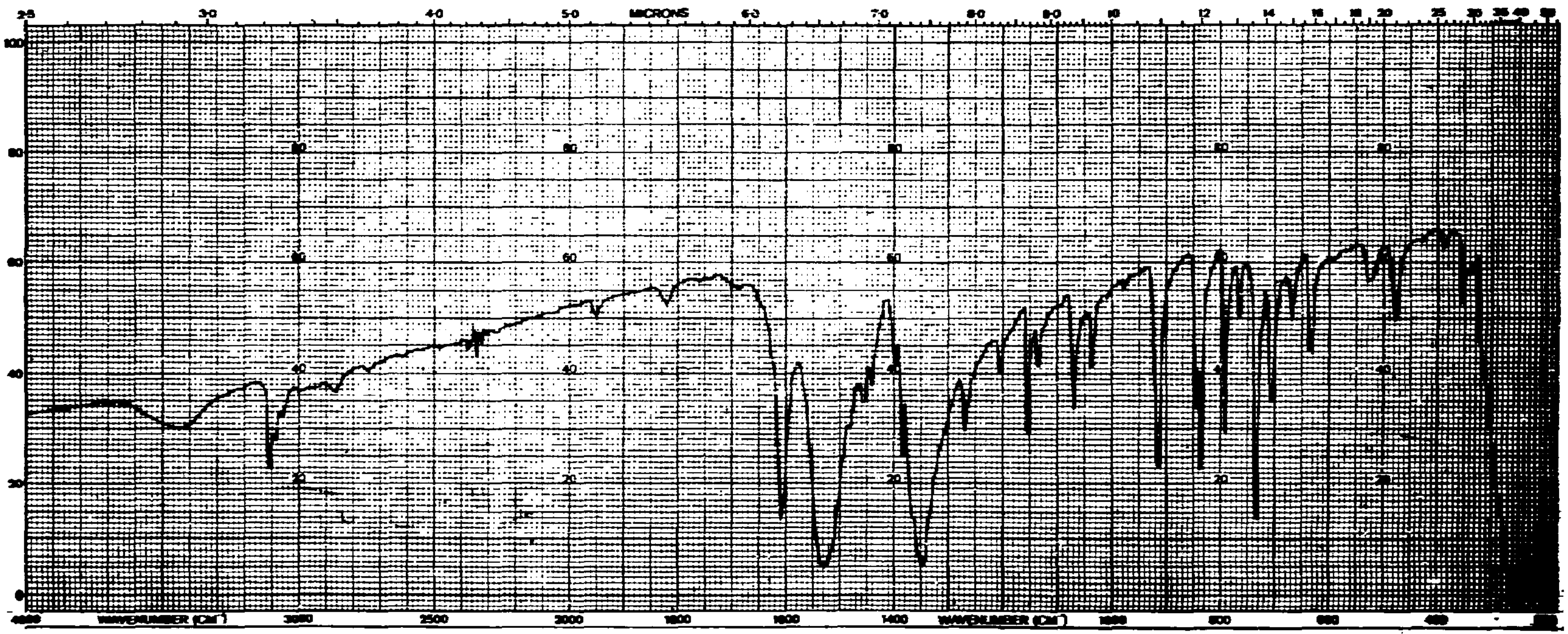


Figure 3. Infrared spectrum of SAR-2,4-DNT of 99.09% purity.

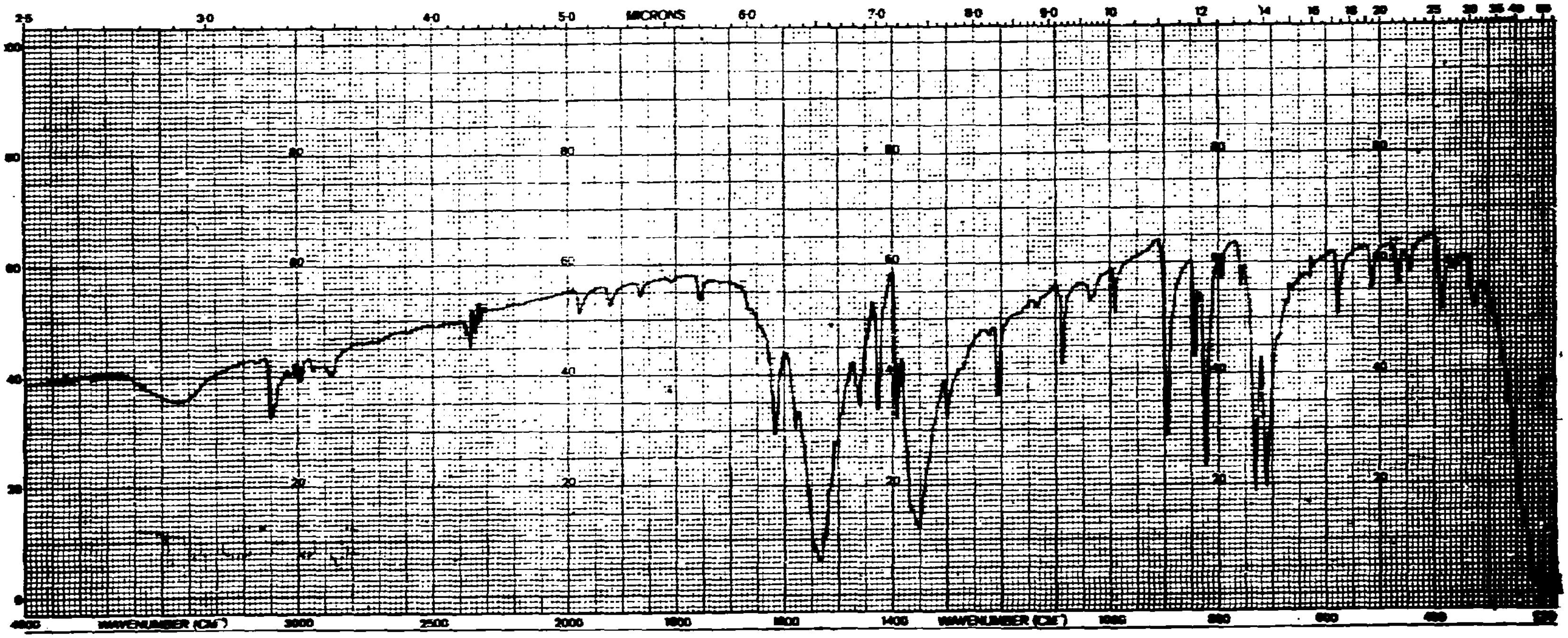


Figure 4. Infrared spectrum of SAR-2,6-DNT of 99.38% purity.

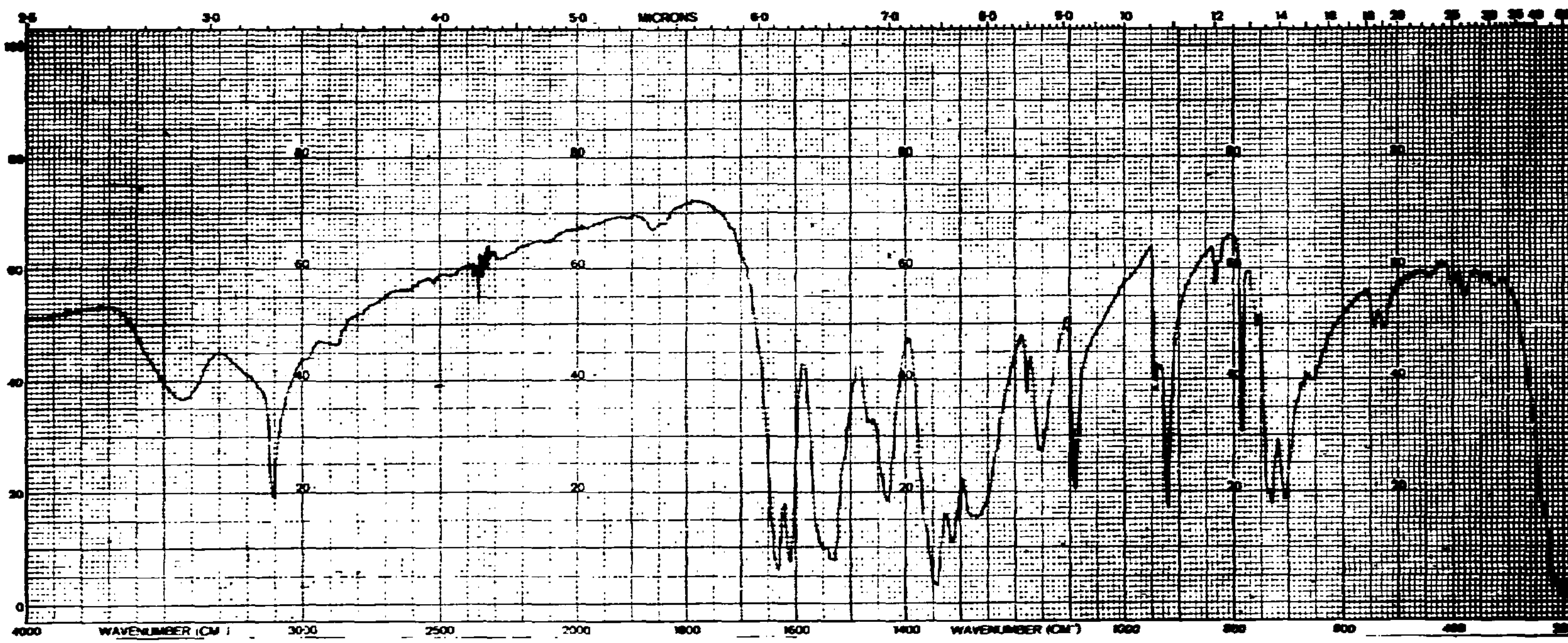


Figure 5. Infrared spectrum of SAR-picric acid of 99.84% purity.

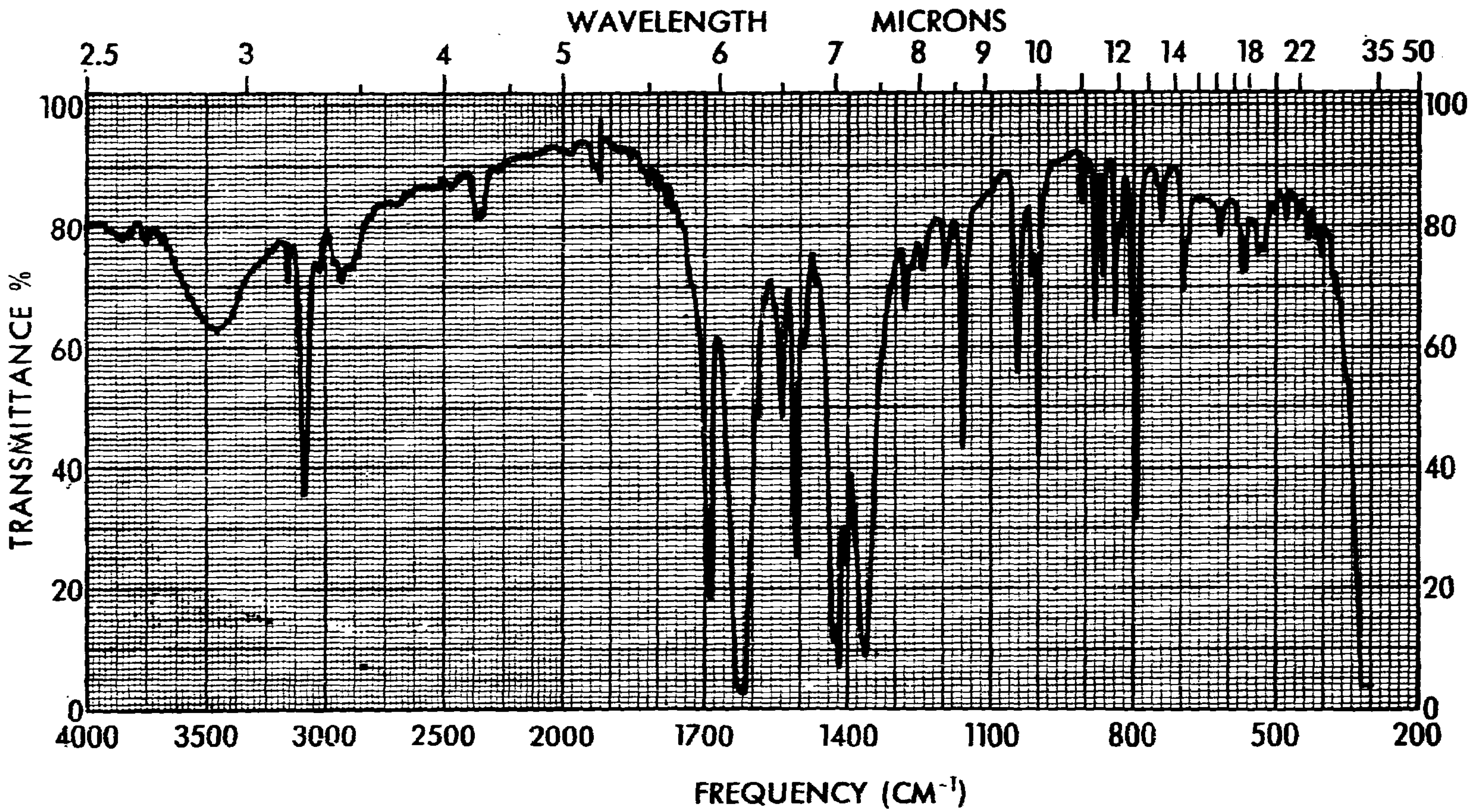


Figure 6. Infrared spectrum of SAR-tetryl of 99.68% purity.

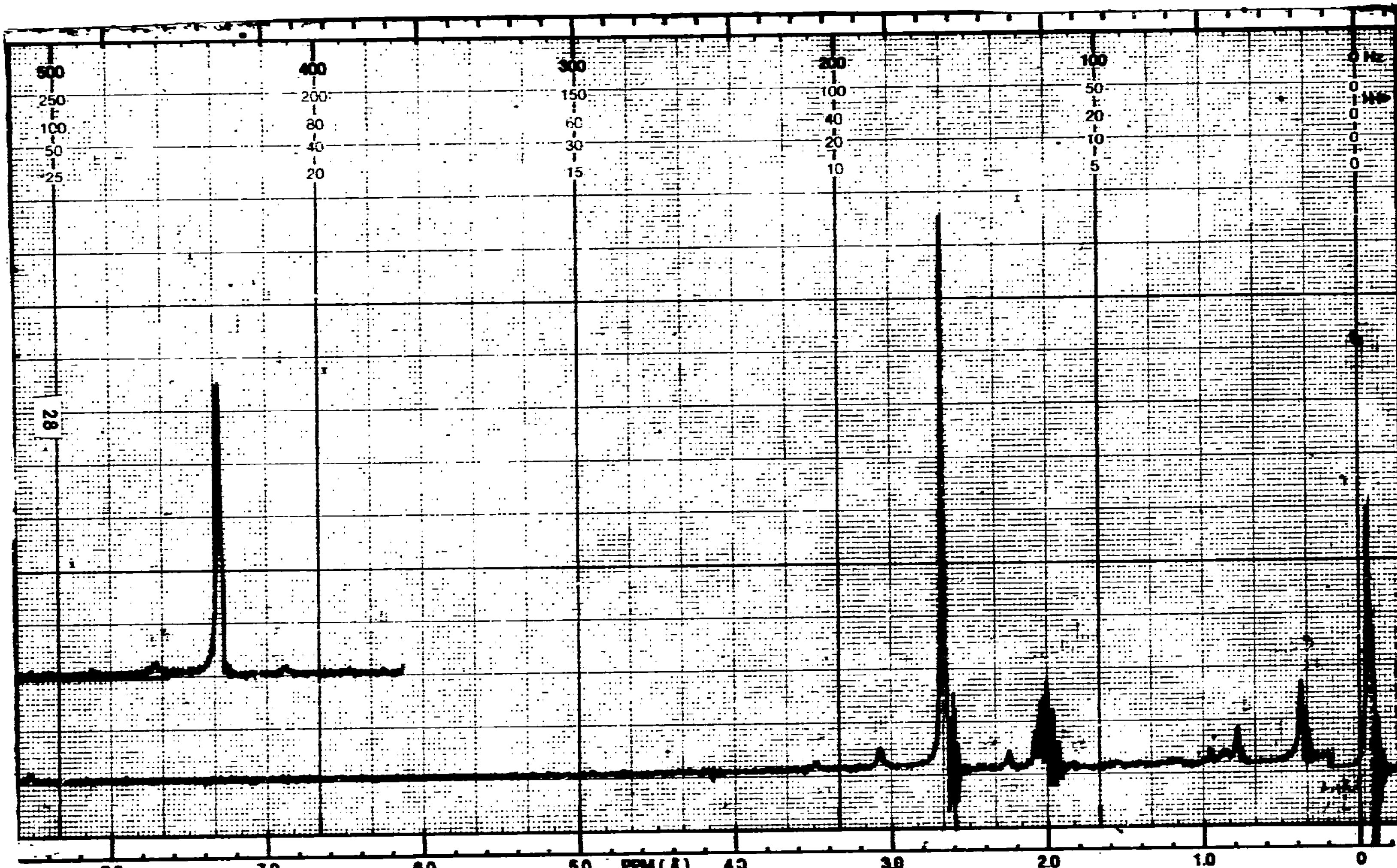


Figure 7. NMR spectrum of SAR-2,4,6-TNT, 99.5% purity, before storage at 70°C.

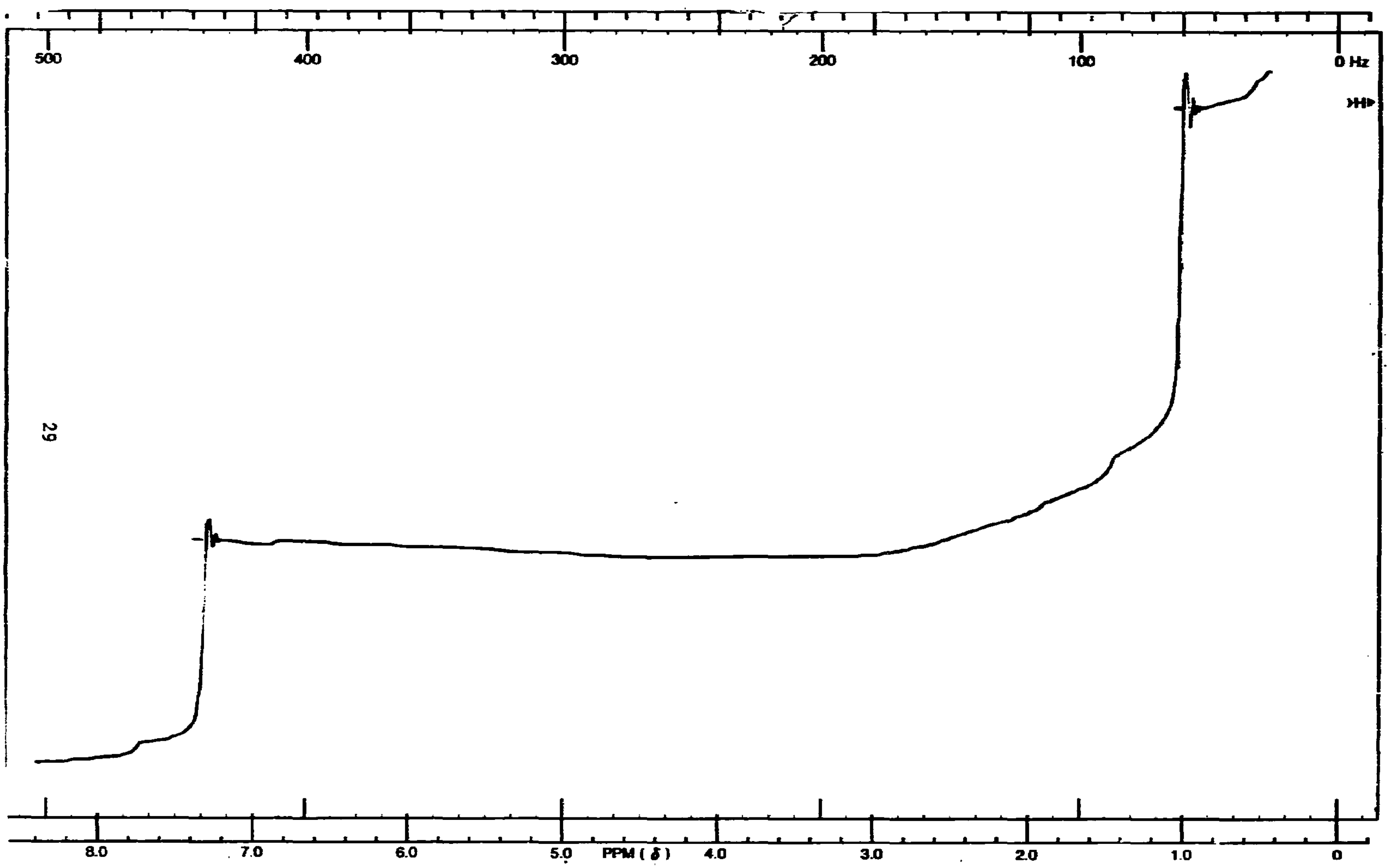
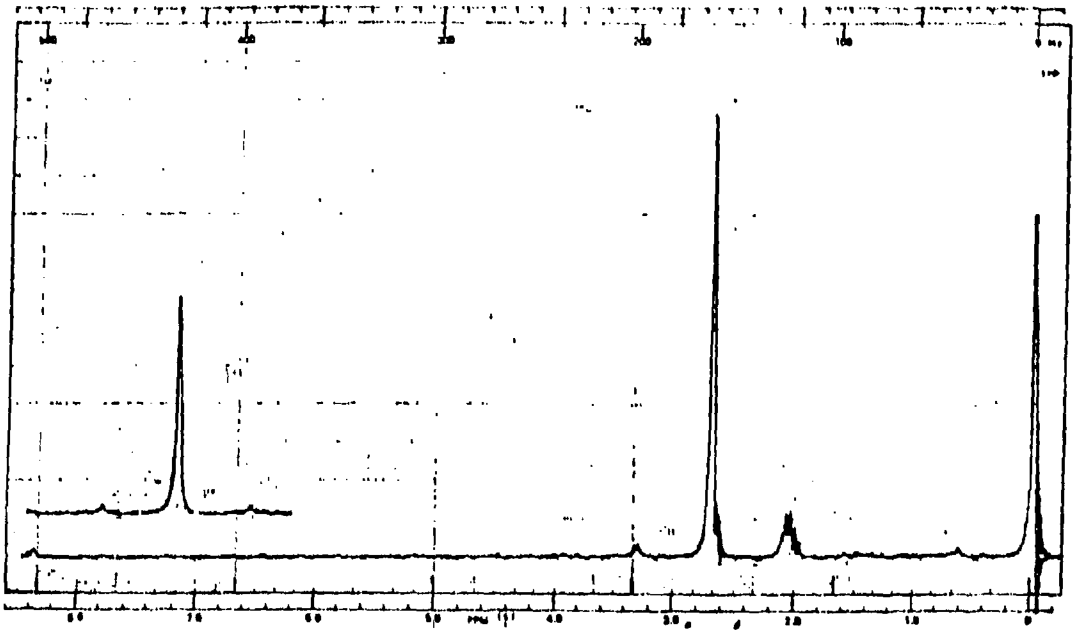
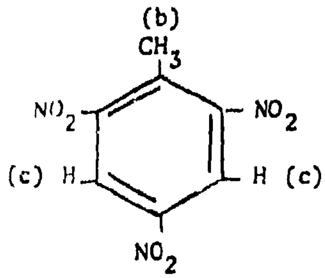


Figure 8. Integral of NMR spectrum of SAR-2,4,6-TNT, 99.5% purity, before storage at 70°C.



Spectrum 23   2,4,6-Trinitrotoluene, Military Grade



Assignments (Hz)

a	122 (impurity)
b	162
c	540

Solvent	d-acetone
Solution filtered	no
Sweep time (sec)	250
Sweep width (Hz)	500
Sweep offset (Hz)	110
RF power level	065
Spectrum amplitude	10
Filter factor	1
Sample spinning rate (RPS)	37

Figure 9. NMR spectrum of 2,4,6-TNT from Picatinny Arsenal Technical Report 4790.

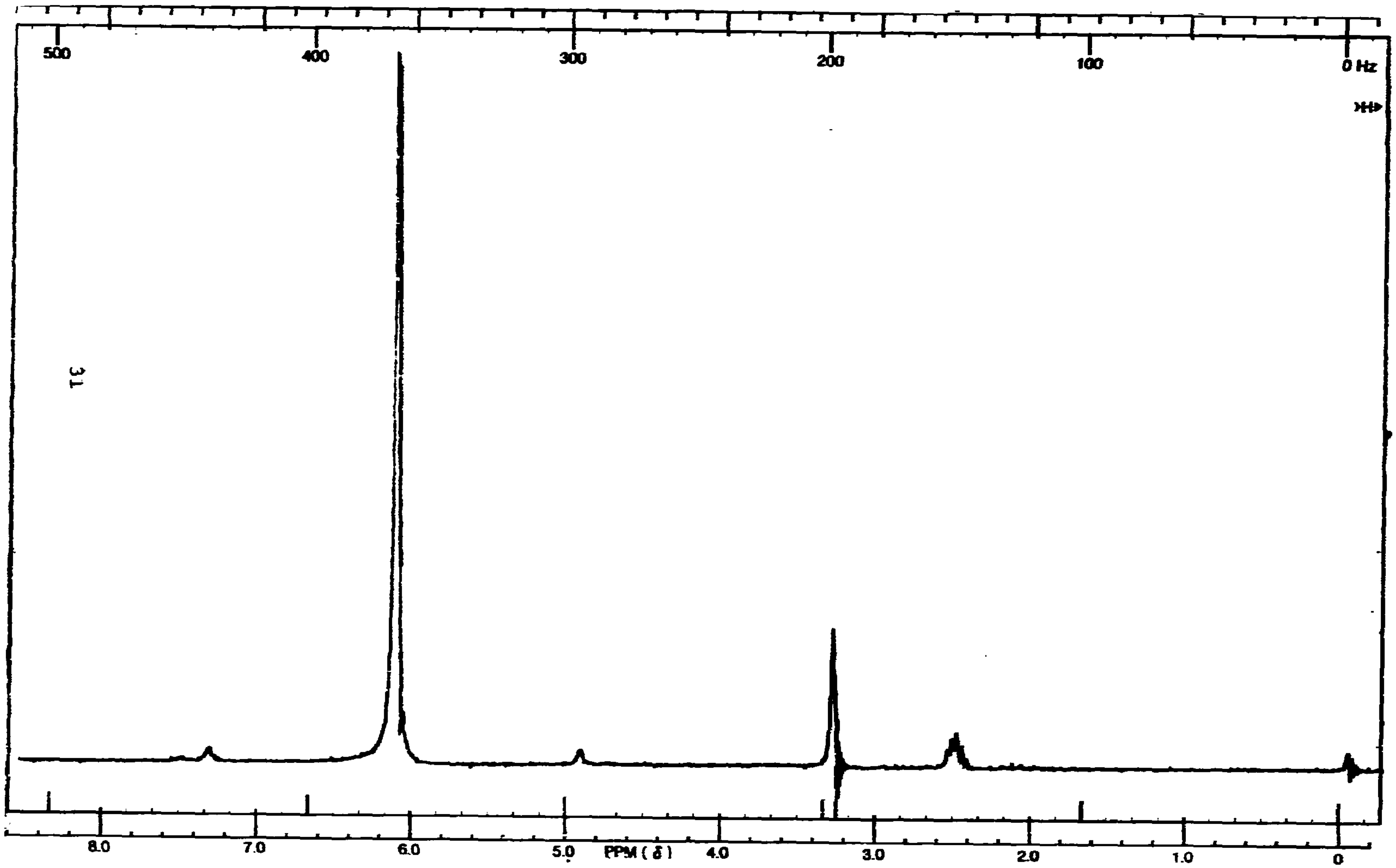


Figure 10. NMR spectrum of SAR-RDX, *dissolved* in d-DMSO, 99.84% purity, before storage at 70°C.

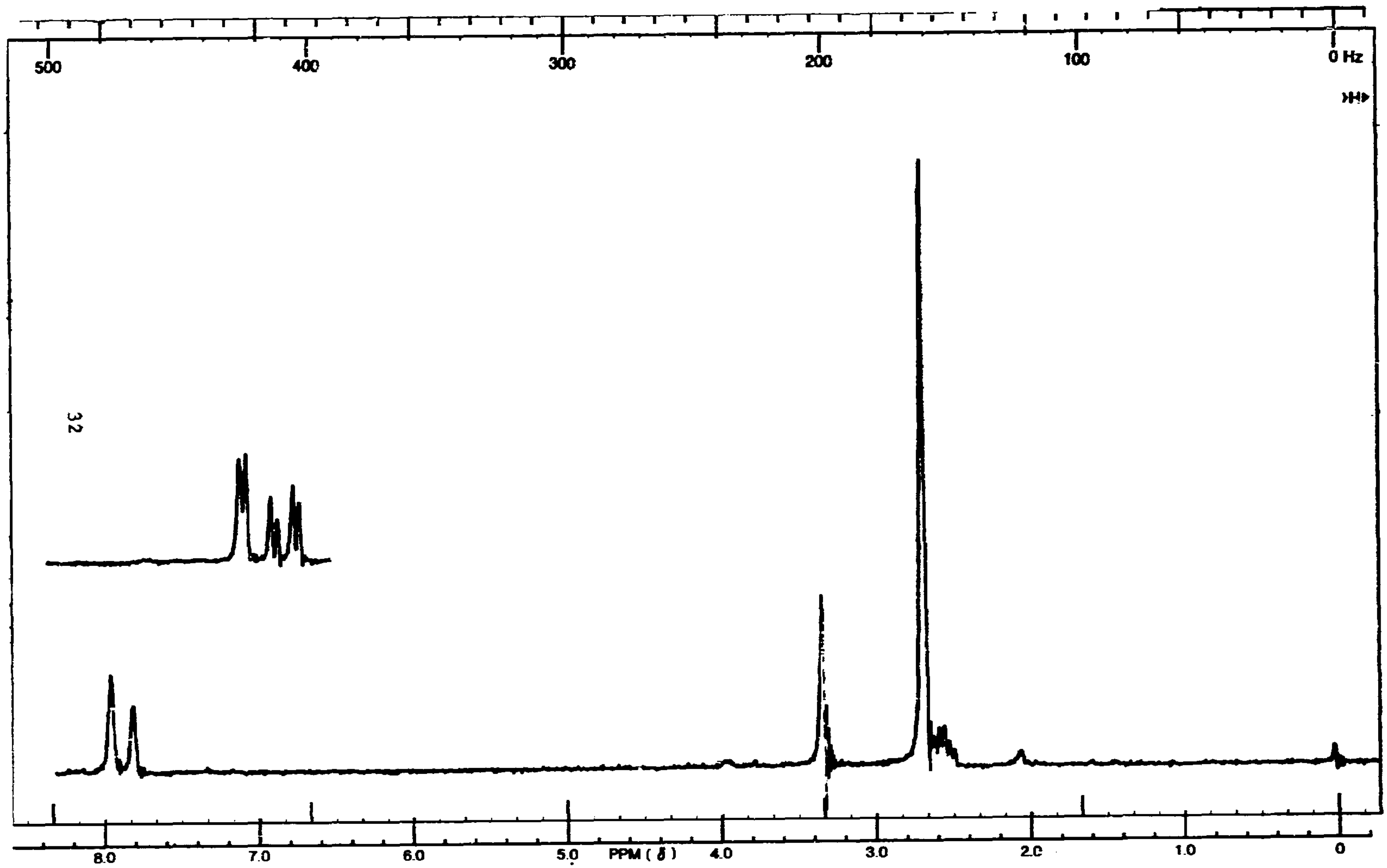


Figure 11. NMR spectrum of SAR-2,4-DNT, dissolved in d-DMSO, 99.09% purity, before storage at 70°C.

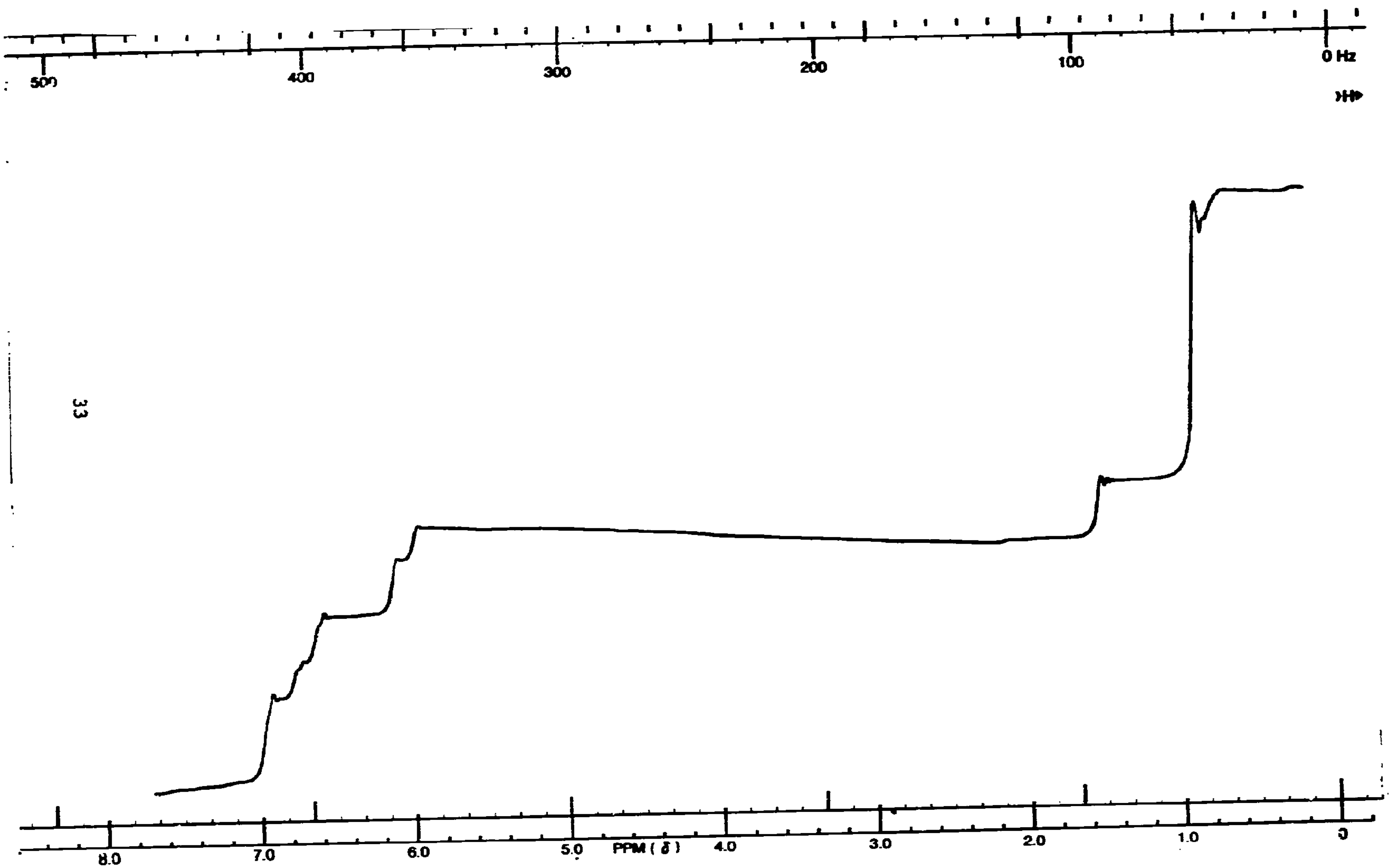


Figure 12. Integral of NMR spectrum of SAR-2,4-DNT, dissolved in d-DMSO, 99.09% purity, before storage at 70°C.

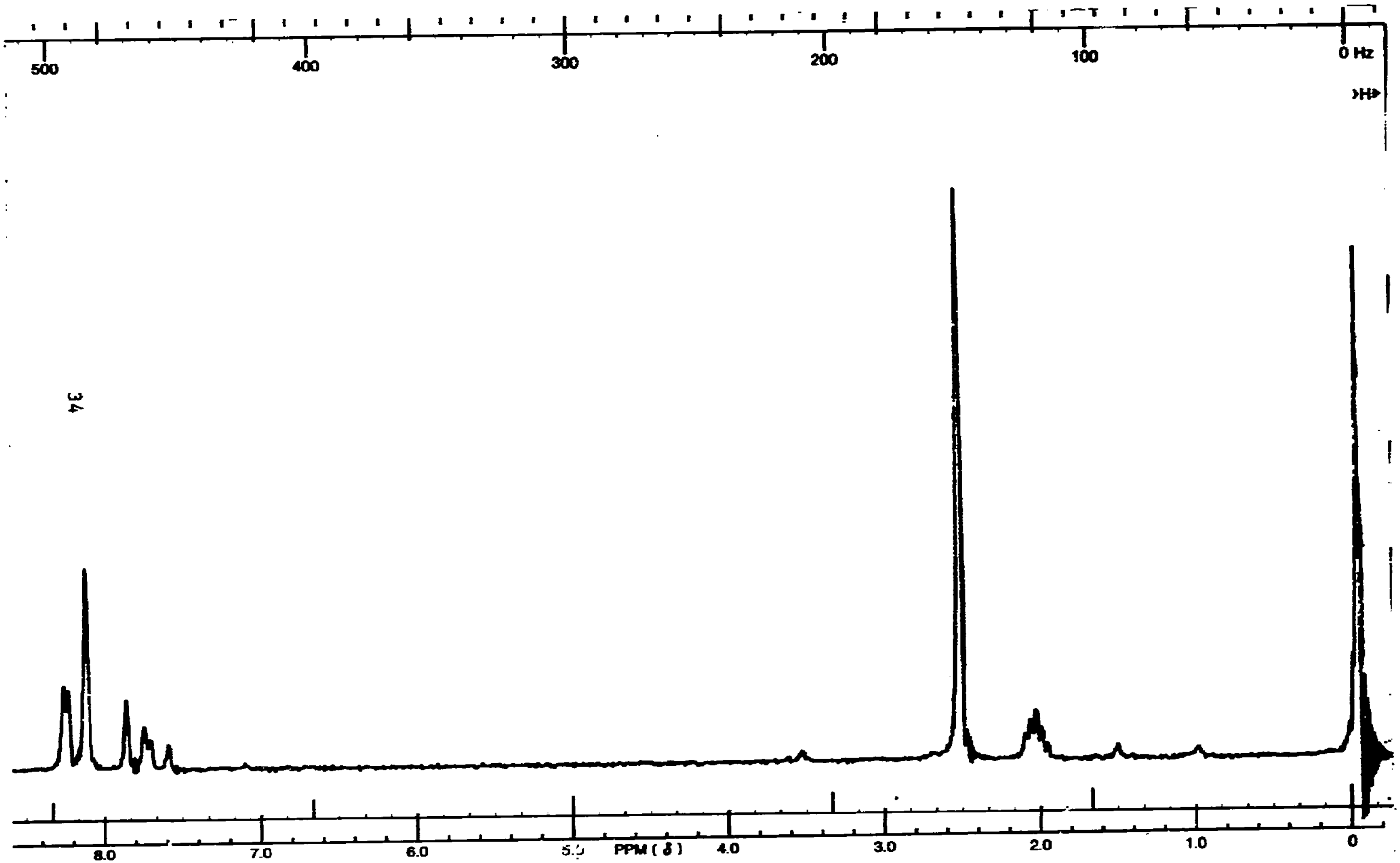


Figure 13. NMR spectrum of SAR-2,6-DNT, dissolved in d-acetone, 99.39% purity, before storage at 70°C.

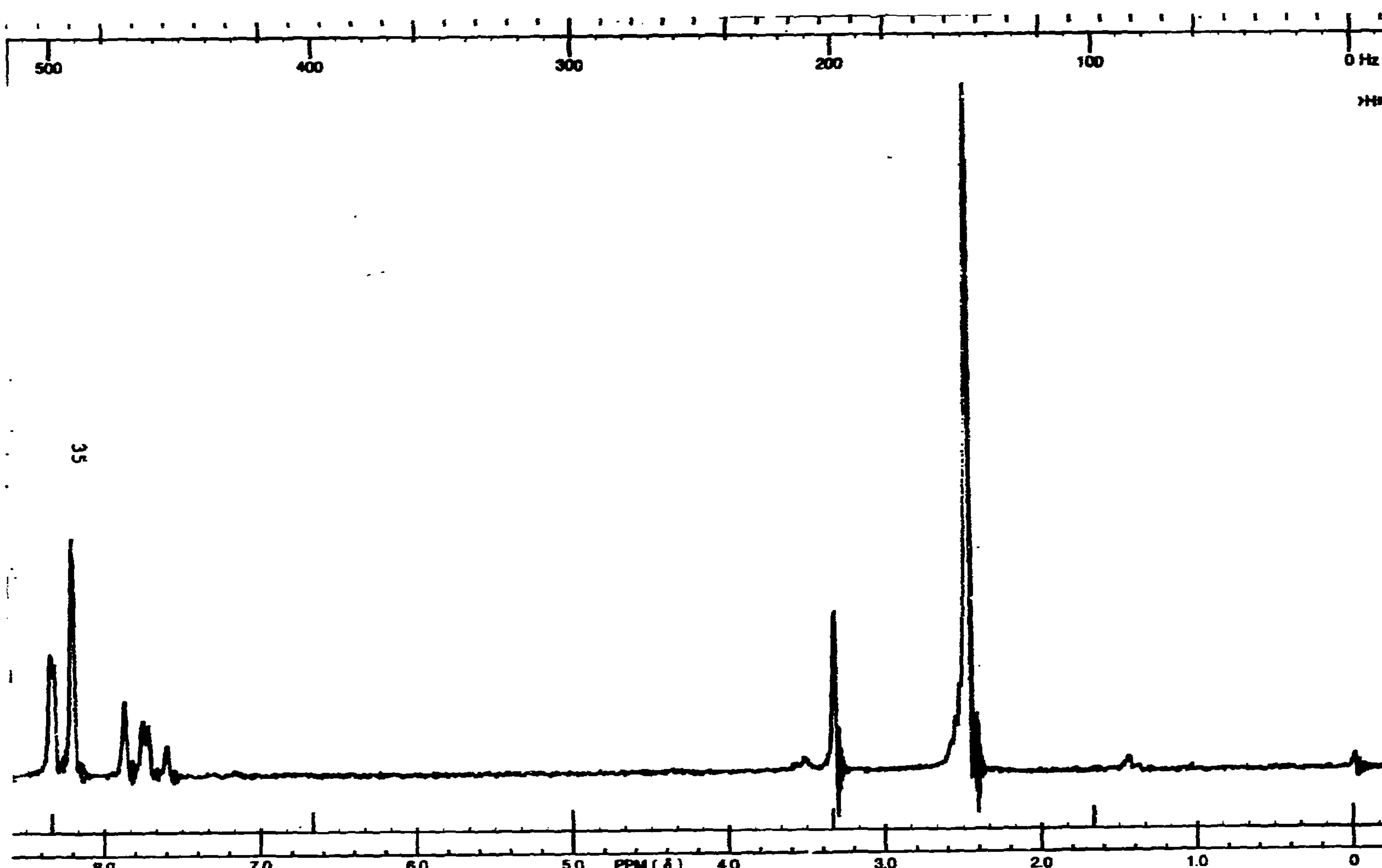


Figure 14. NMR spectrum of SAR-2,6-DNT, dissolved in d-DMSO, 99.39% purity, before storage at 70°C.

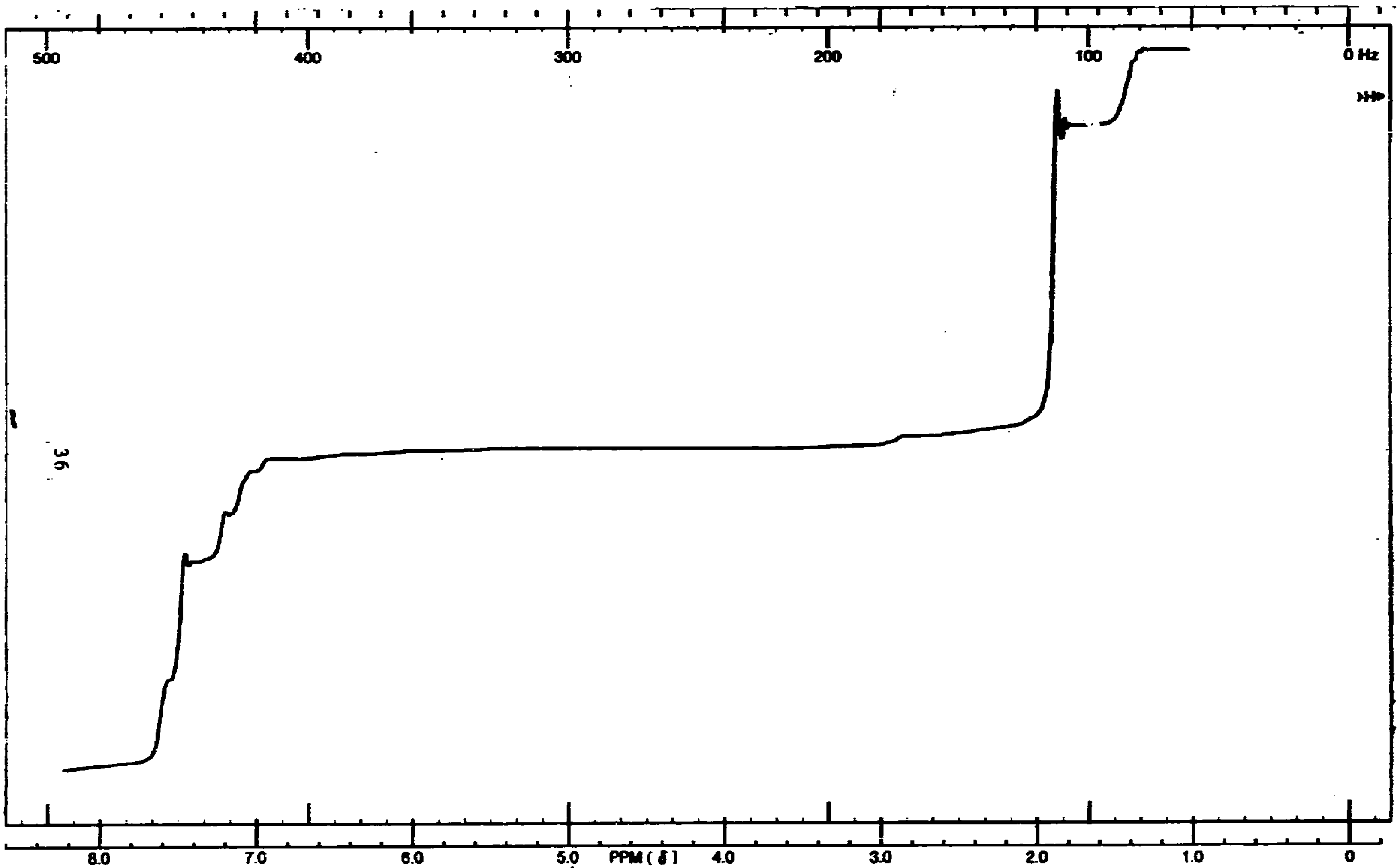


Figure 15. Integral of NMR spectrum of SAR-2,6-DNT, dissolved in d-acetone, 99.39% purity, before storage at 70°C.

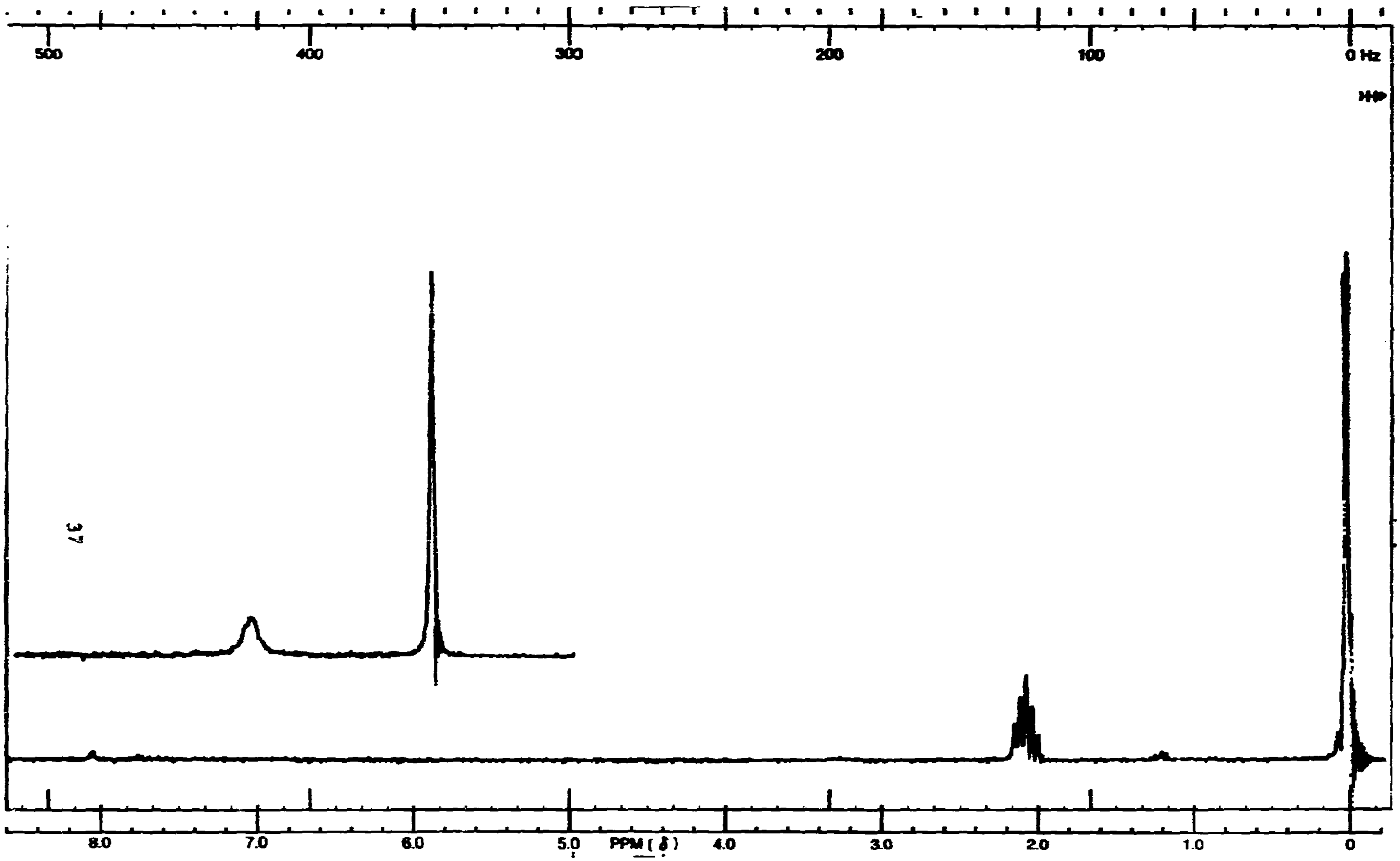


Figure 16. NMR spectrum of SAR-picric acid, dissolved in d-acetone, 99.89% purity, before storage at 70°C.

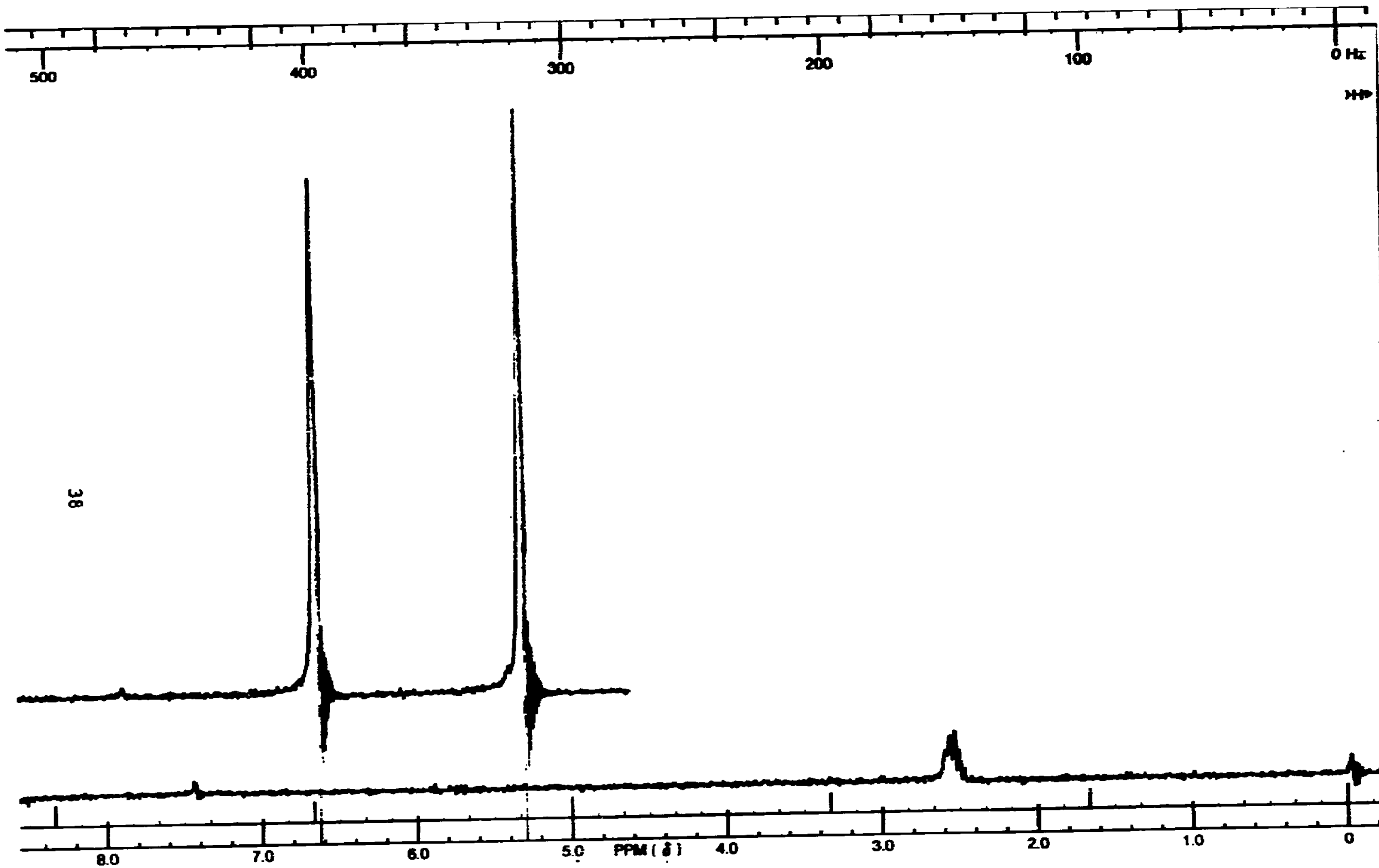


Figure 17. NMR spectrum of SAR-picric acid, dissolved in d-DMSO, 99.89% purity, before storage at 70°C.

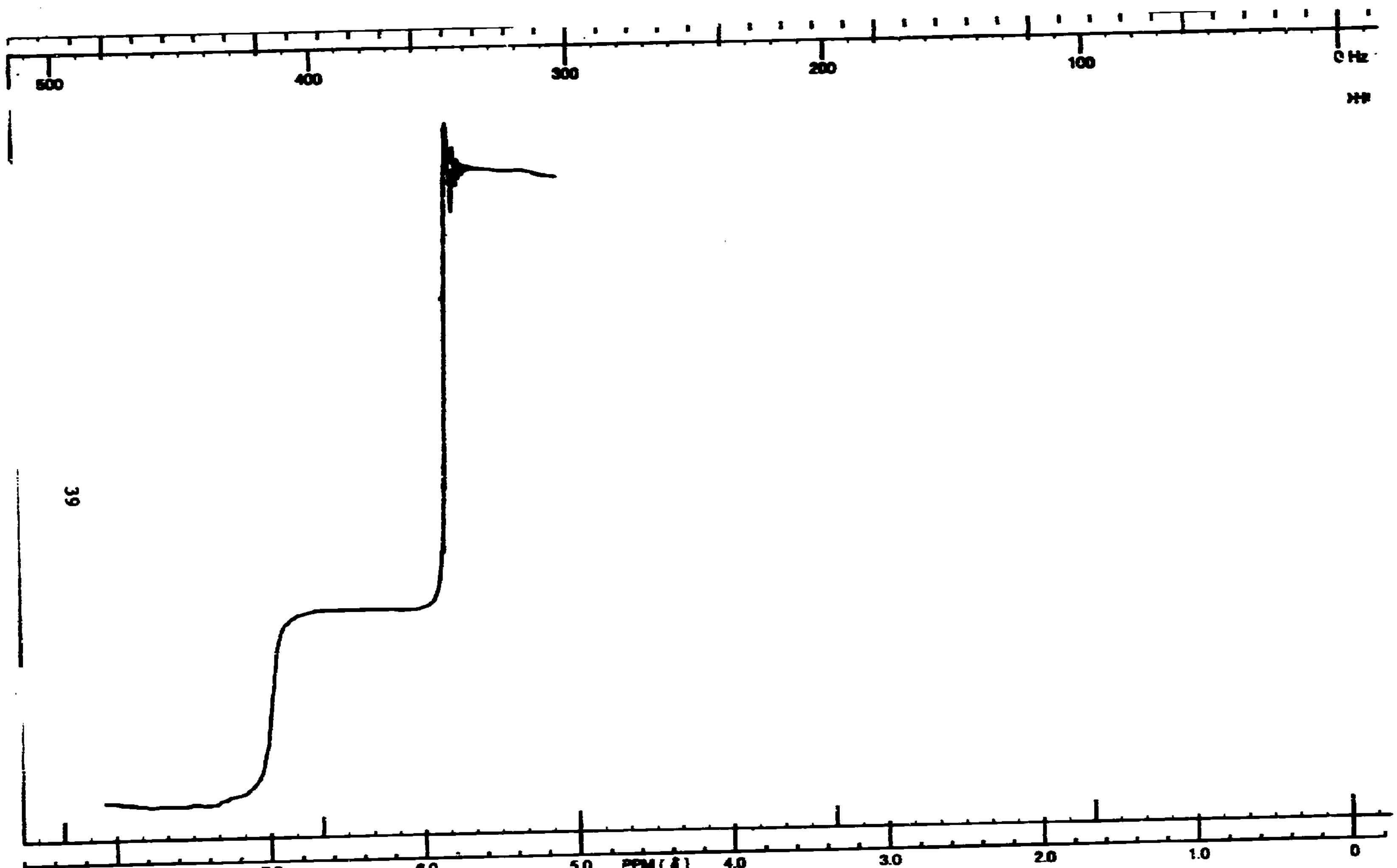


Figure 18. Integral of NMR spectrum of SAR-picric acid, dissolved in d-acetone, 99.89% purity, before storage at 70°C.

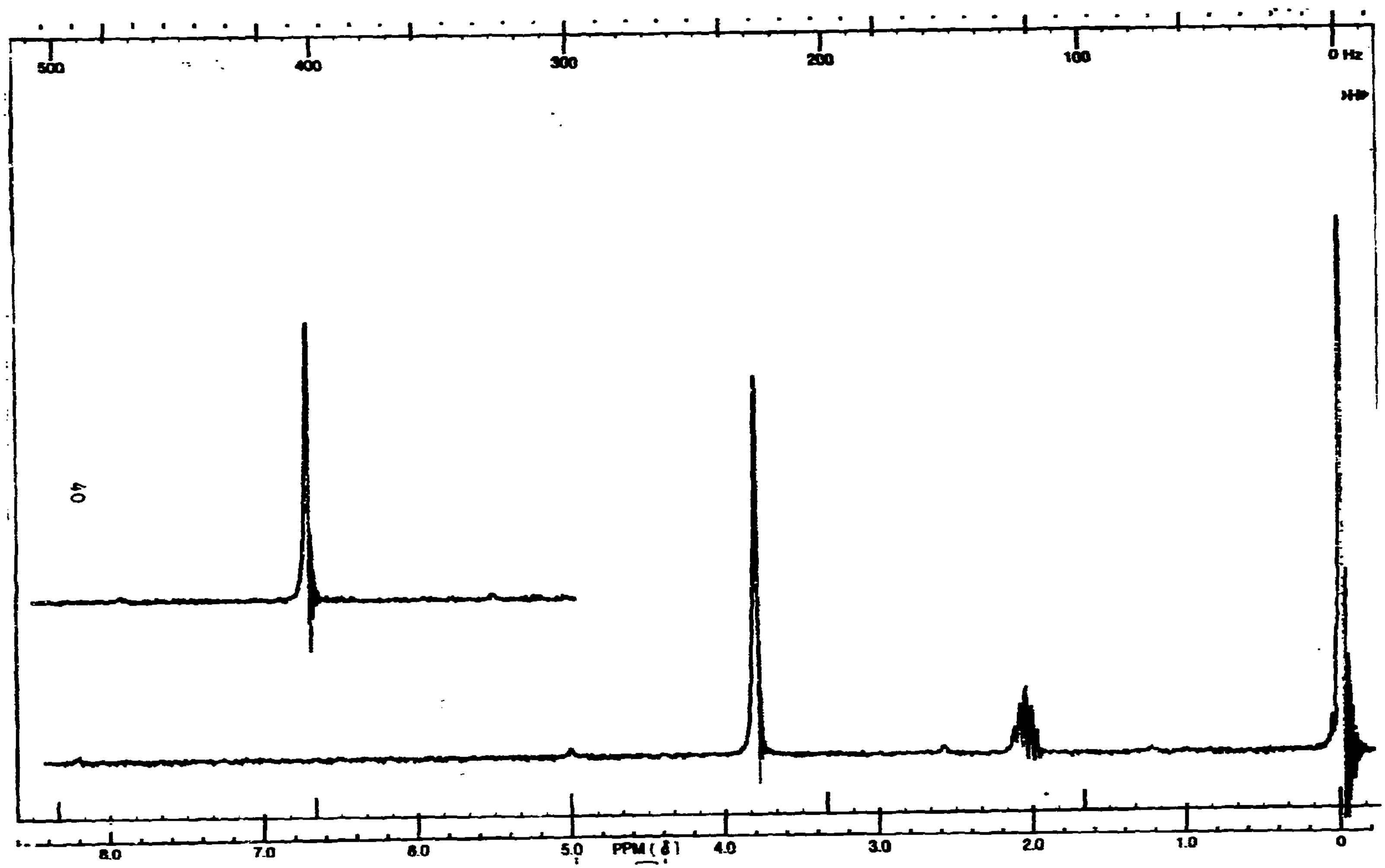


Figure 19. NMR spectrum of SAR-tetryl, 99.68% purity, dissolved in d-acetone, before storage at 70°C.

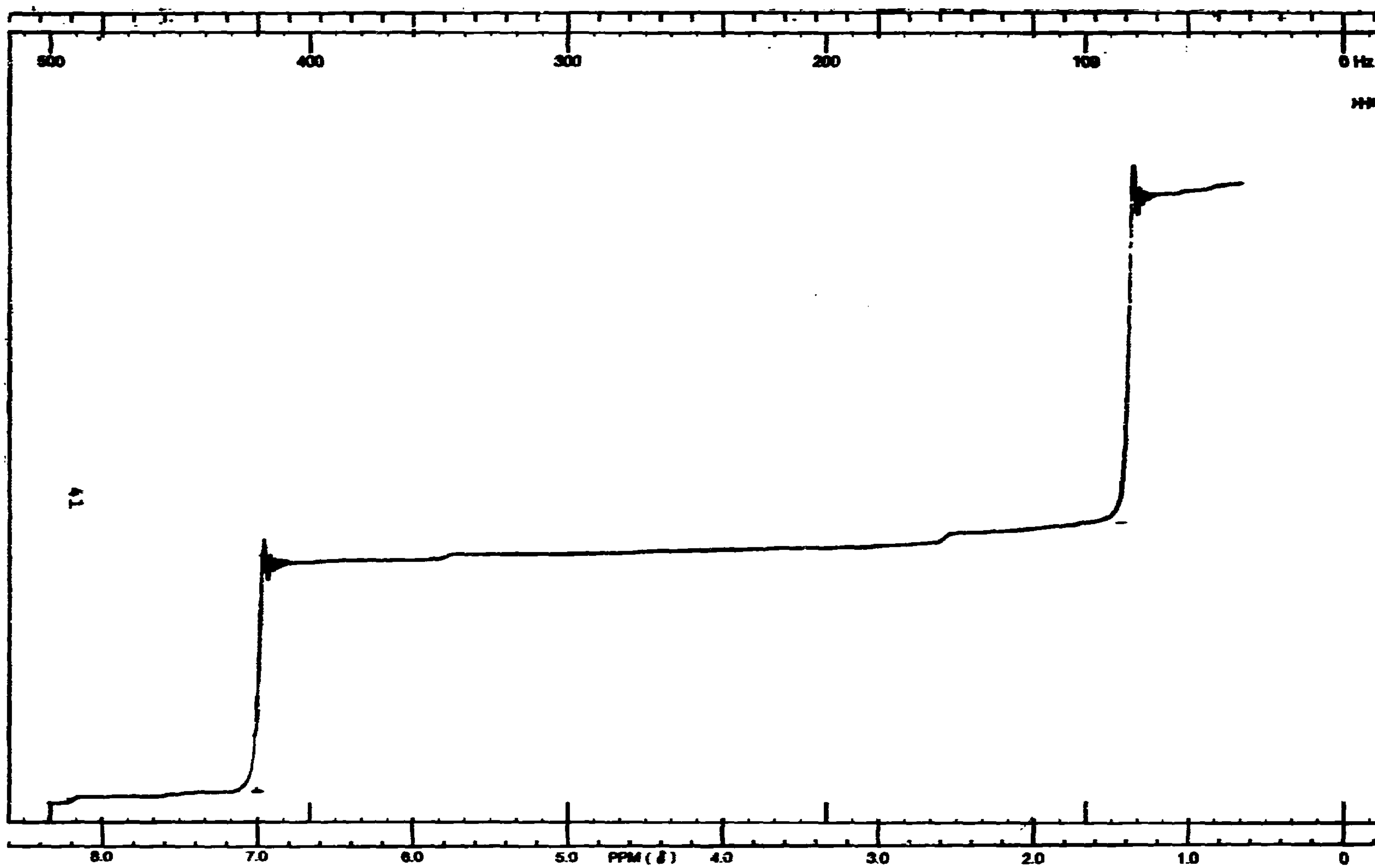


Figure 20. Integral of NMR spectrum of SAR-tetryl, 99.71% purity, dissolved in d-acetone, before storage at 70°C.

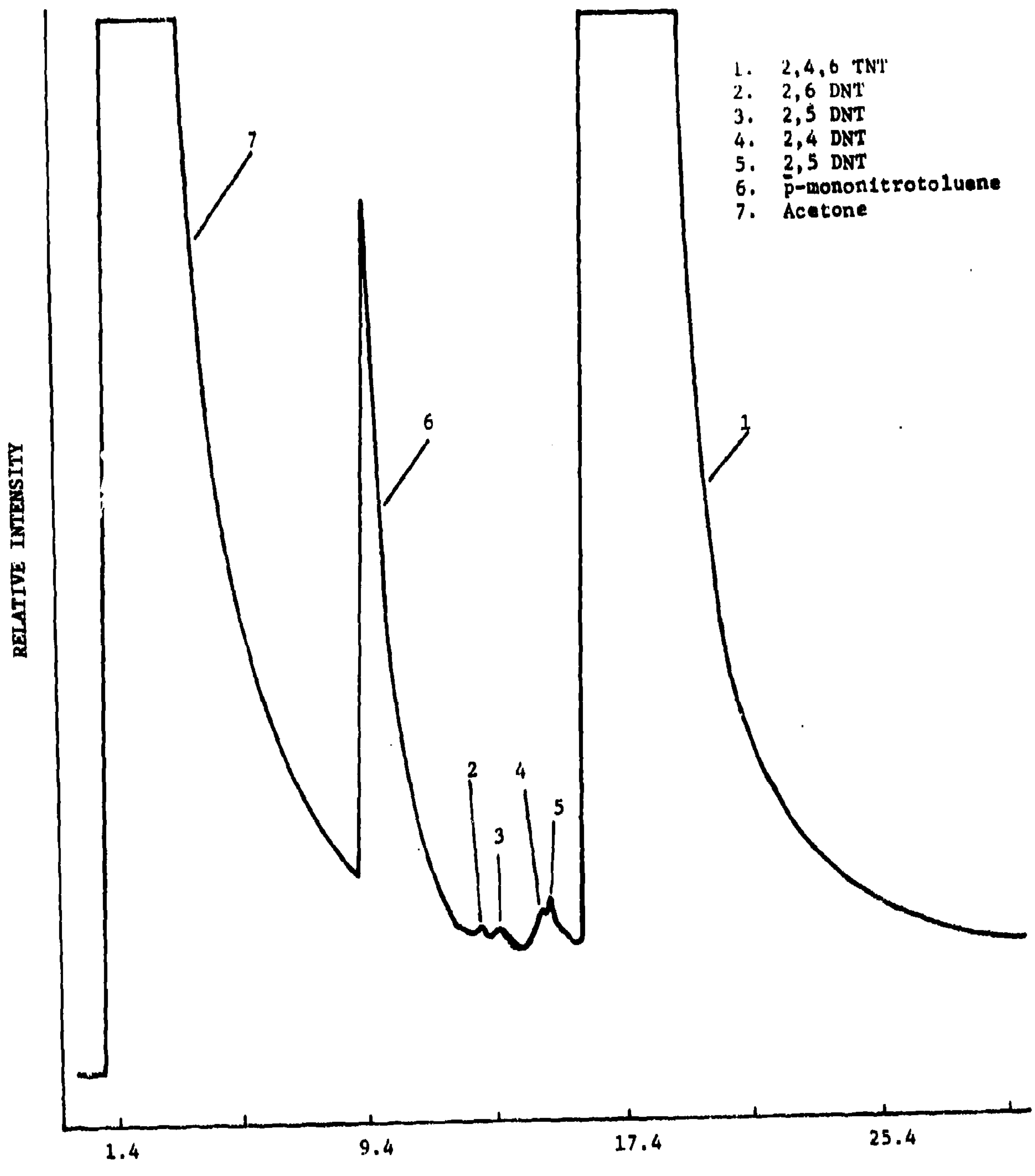


Figure 21. GC of SAR-2,4,6-TNT purity before storage at 70°C.

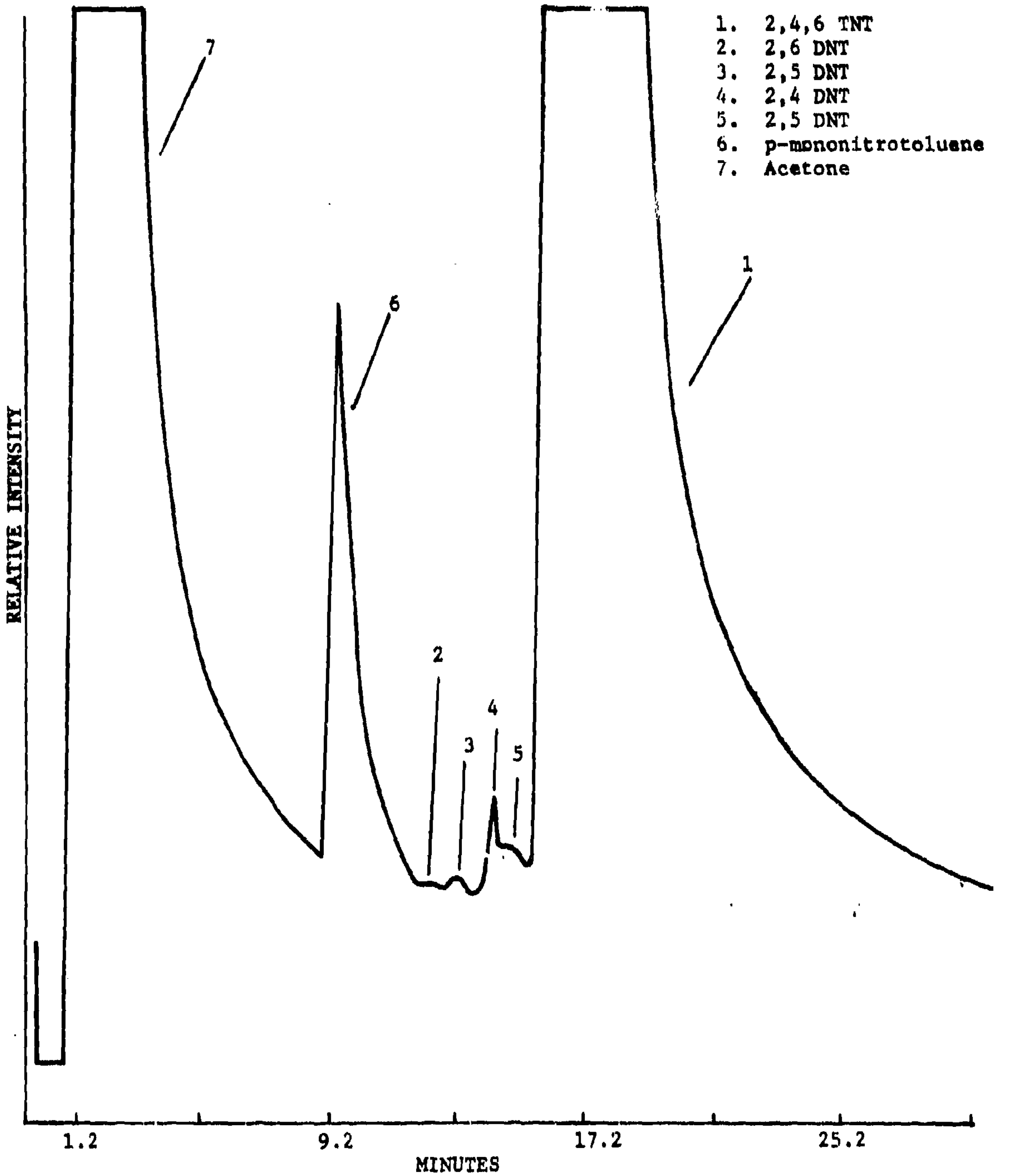


Figure 22. GC of SAR-2,4,6-TNT, 99.5% purity, after storage at 70°C.

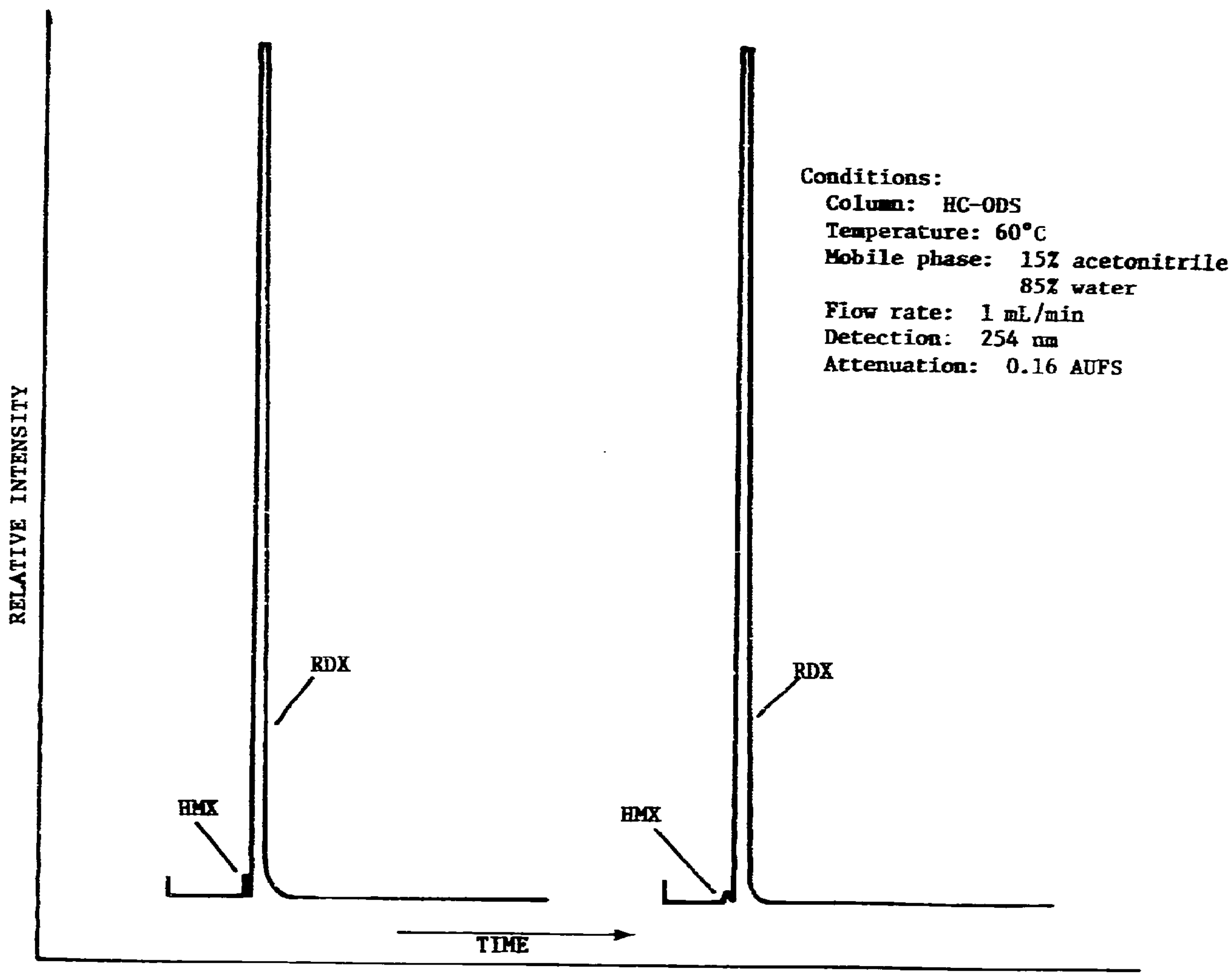


Figure 23. LC of SAR-RDX before (right) and after (left) 2 weeks' storage at 70°C.

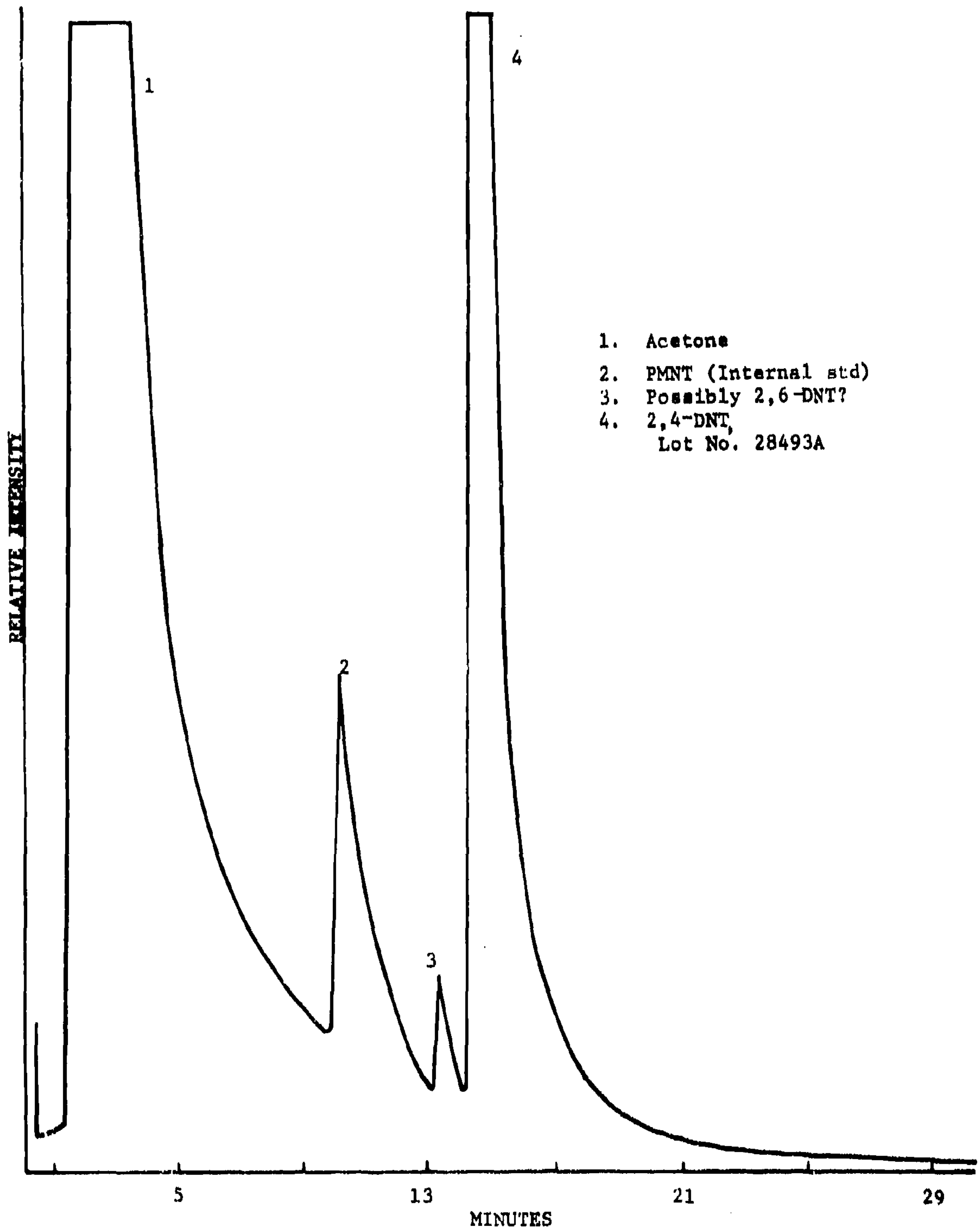


Figure 24. GC of SAR-2,4-DNT, 99.1% purity before storage at 70°C.

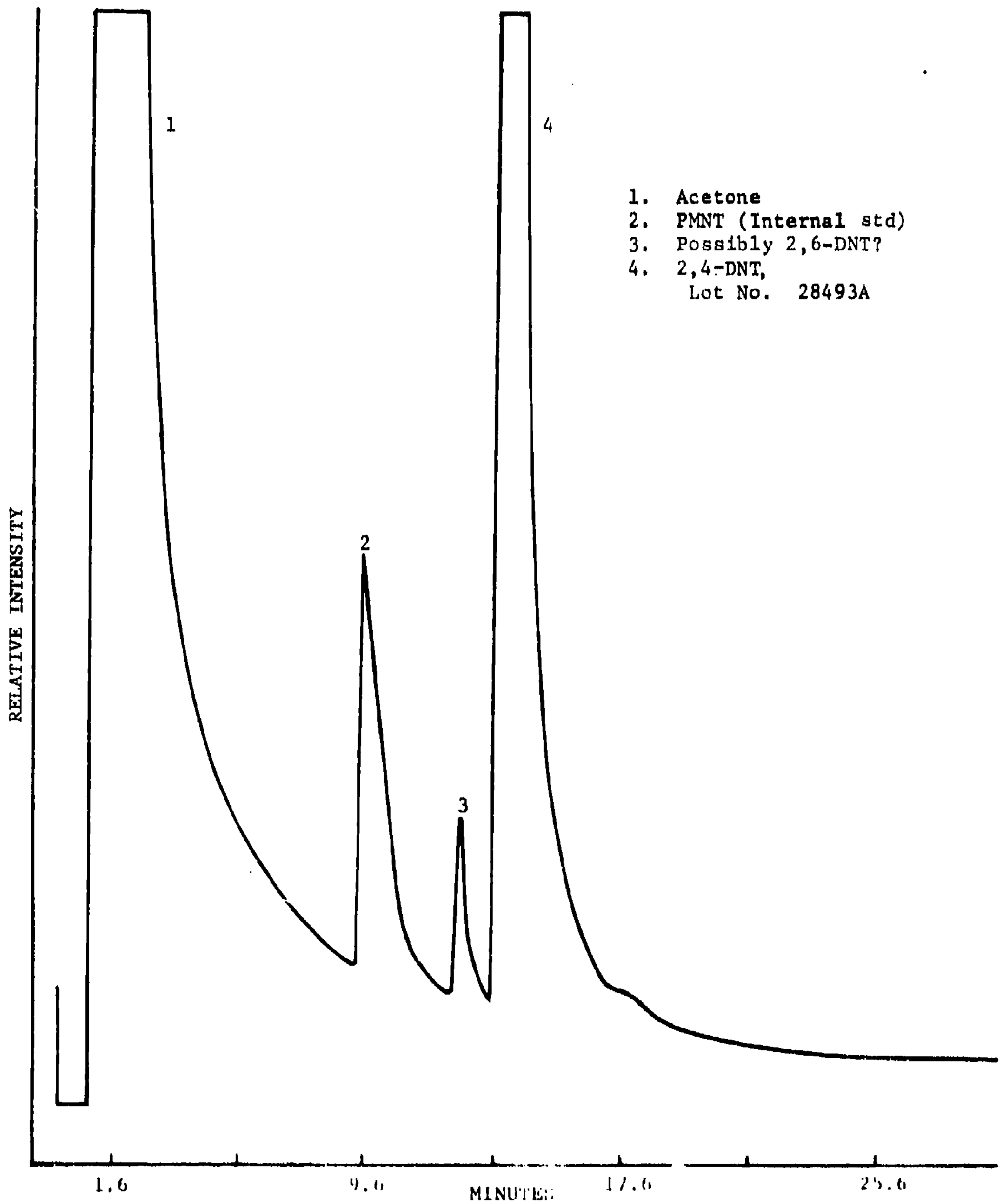


Figure 25. GC of SAR-2,4-DNT, 99.2% purity after storage at 70°C.

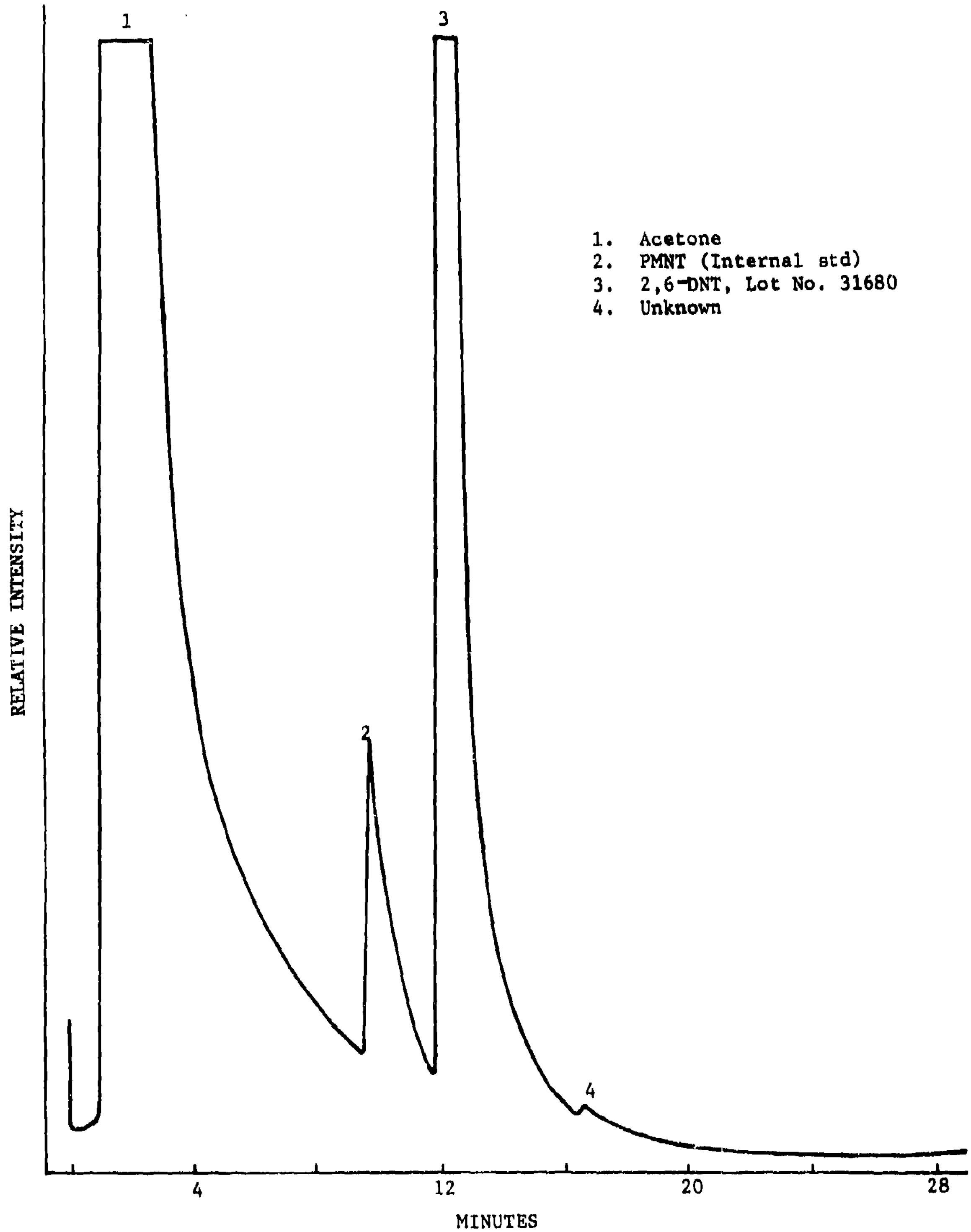


Figure 26. GC of SAR-2,6-DNT, 99.4% purity before storage at 70°C.

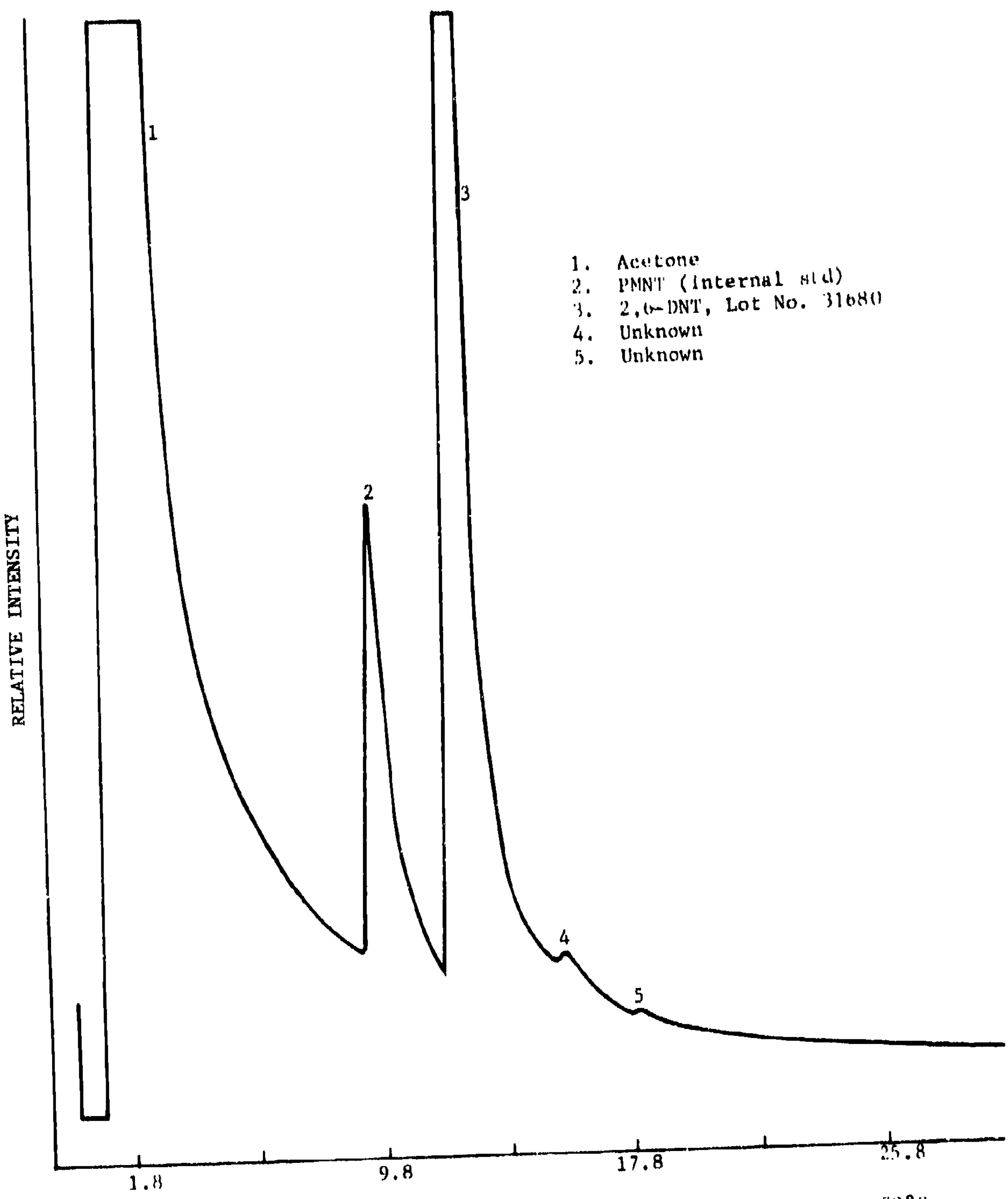


Figure 27. GC of SAR-2,6-DNT, 99.4% purity after storage at 70°C.

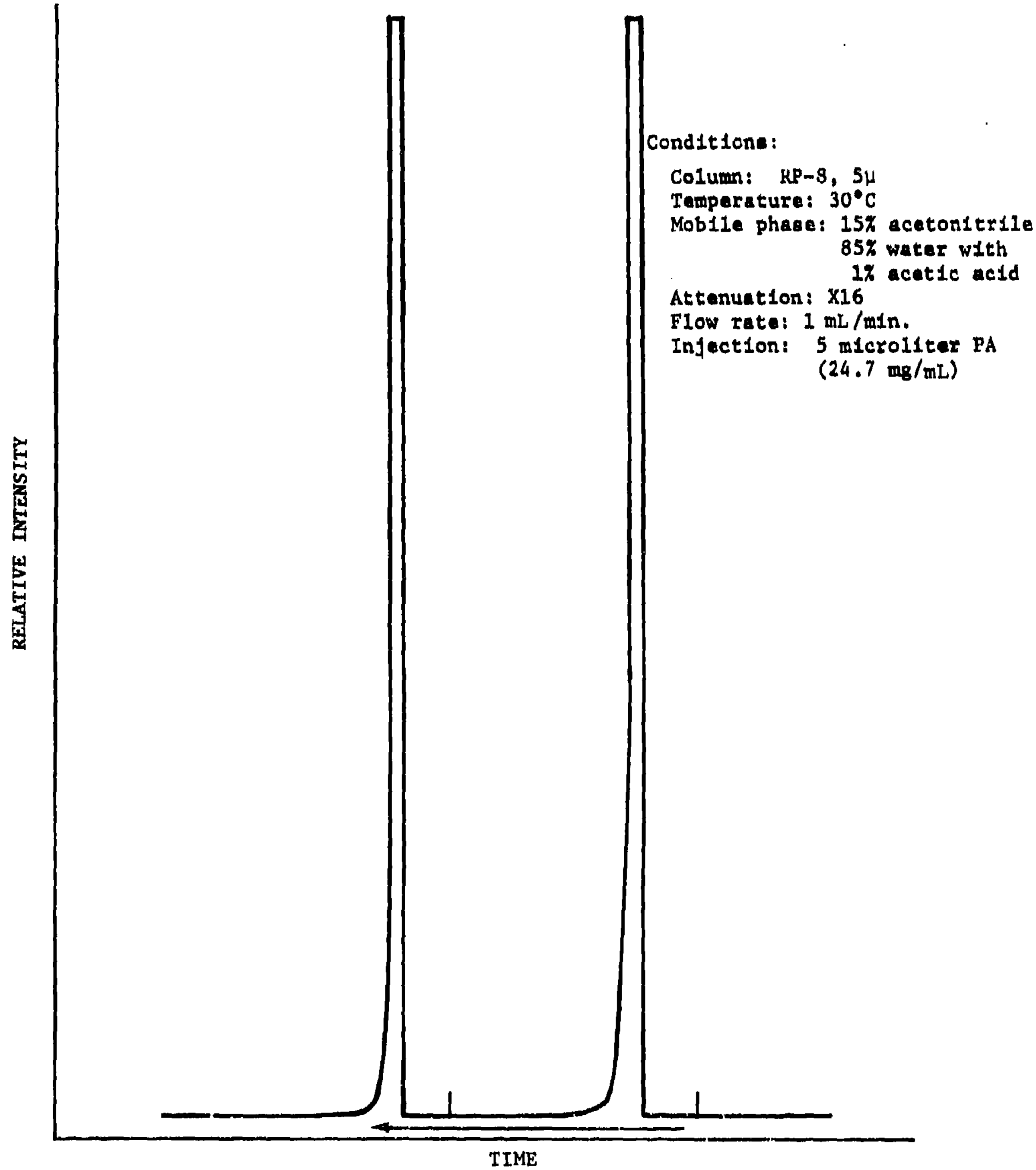


Figure 28. LC of SAR-picric acid before (right) and after (left) 2 weeks storage at 70°C

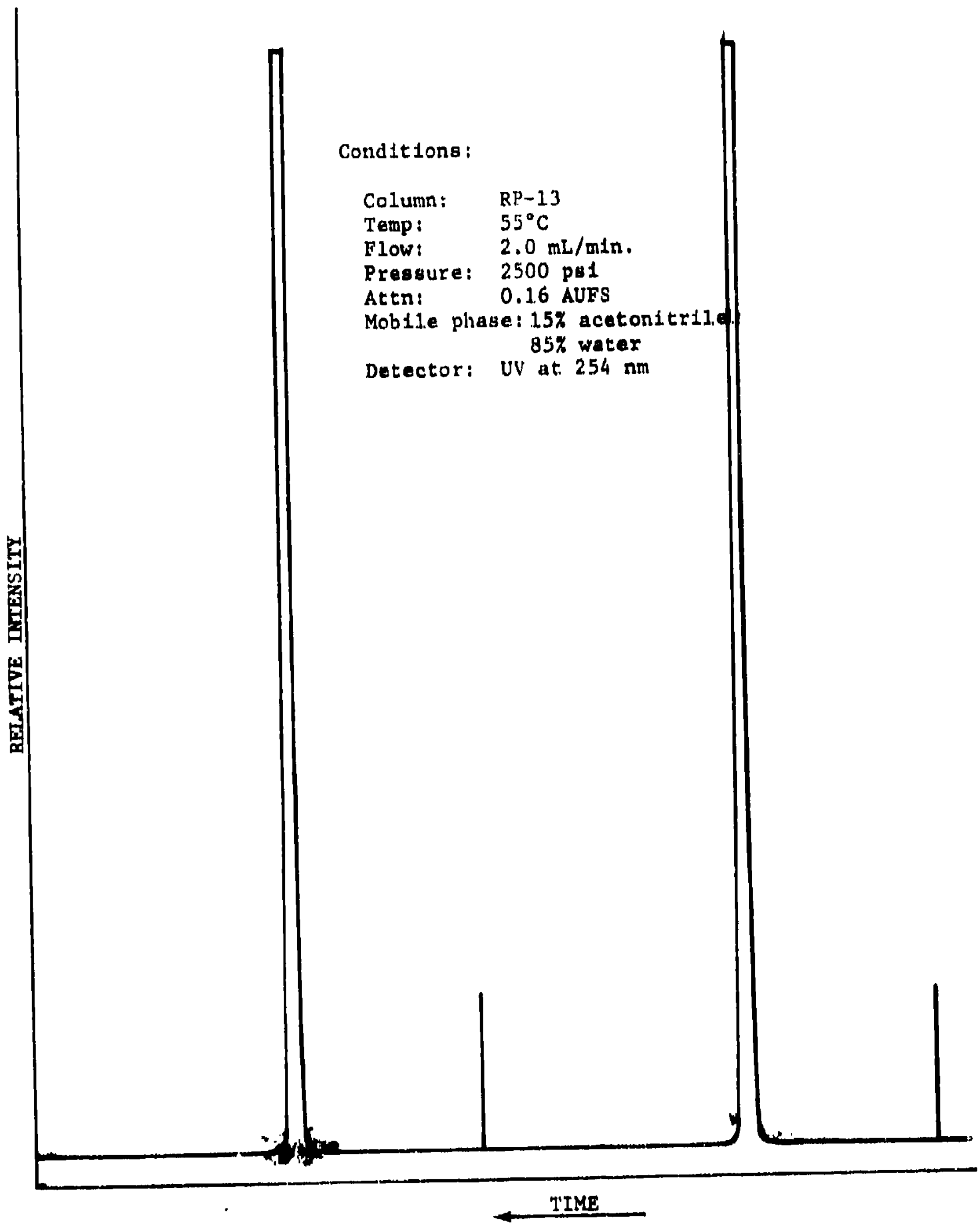


Figure 29. LC of SAR-tetryl before (right) and after (left) 2 weeks storage at 70°C.

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MEMORANDUM REPORT ARLCD-MR-79002

QUALIFICATION OF ANALYTICAL REFERENCE ENERGETIC MATERIALS

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